Journal of Sustainable Agriculture And the Environment

E-mail: jsae@mouau.edu.ng. Website: www.mouau.edu.ng.

Comparison of Different Methods for the Assessment of Soil Organic Carbon in Northern Guinea Savanna Zone of Nigeria

Mustapha A. A.

Department of Soil Science, Bayero University Kano, Kano, Nigeria

aamustapha.ssc@buk.edu.ng. +2348034751783

Abstract

In Nigeria, Walkley-Black is the most widely used method for soil organic carbon determination. The organic carbon content of the Northern Guinea Savanna soil is low and incomplete oxidation of carbon occurs in the method of determination (Walkley-Black). Three different methods; Tube digestion, Loss on ignition and Potassium permanganate were evaluated and the results obtained compared with Walkley-Black. Soil were sampled from the Northern Guinea Savanna of Nigeria and amount of carbon evaluated using the aforementioned methods. Percentage of carbon recovered ranged from 50.637 to 132.059 and correction factor (CF) varied from 0.757 to 1.975. The result showed low correlation among the used methods.The highest and lowest coefficient of determination (R^2) was found in Tube digestion (0.113) and loss on ignition methods (0.014) respectively. The results of the recovery percentages indicates that the loss on ignition and tube digestion were almost similar with the results obtained using the Walkey-Black and as such may be used as an alternative to Walkley-Black method in soil testing laboratories for soils of the Northern Guinea Savanna of Nigeria.

Introduction

Soil organic carbon (SOC) is essential for soil and environmental quality. It is a key component of soil organic matter (SOM), affecting physical, chemical and biological properties of the soil and it plays a critical role in the global carbon balance and thought to be a major factor in global climate change (Roper *et al.,* 2019). Better understanding of soil organic carbon can help evaluate and classify soils, and assist in application of best management practices for irrigation, fertilization and pesticide application (Strosser, 2010). Soil organic carbon is one part in the much larger global carbon cycle that involves the cycling of carbon through the soil, vegetation, ocean and the atmosphere.

In principle, the quantity of soil organic carbon stored in a given soil is dependent on the equilibrium between the amount of carbon entering the soil and the amount of carbon leaving the soil as carbon-based respiration gases resulting from microbial mineralization and, to a lesser extent, leaching from the soil as dissolved organic carbon.

Locally, carbon can also be lost or gained through soil erosion or deposition, leading to the redistribution of soil carbon at local, landscape and regional scales. Levels of soil organic carbon storage are therefore mainly controlled by managing the amount and type of organic residues that enter the soil (i.e. the input of organic carbon to the soil system) and minimizing the soil carbon losses (Idowu *et al.,* 2008; Roper et al., 2019; Weil *et al.,* 2003)

In Nigeria, Walkley-Black method is the most widely used method for soil organic carbon determination, and an argument has been raised pertaining the complete oxidation of carbon as well as the hazardous fumes released during the process of determination due to the use of concentrated sulphuric acid. There is a need to explore other methods of determination in other to achieve complete oxidation as well as production of environmental friendly fumes. This research was carried out to compare different methods of determination of carbon in Dakace and Soba in Northern Guinea Savannah of Nigeria as well as assess the recovery efficiencies of each method

Materials and Method

Soil Sampling and Processing: The soil samples were obtained from Dakace and Soba Areas of Kaduna State. Samples were collected using augers at a distance of 1 km and at a depth of 0-20 cm with a total of 10 samples in each location. The samples were air dried and passed through 2 mm sieve and stored for onward analysis in the laboratory.

Laboratory Analyses.

Each of the prepared samples was analyzed for organic carbon using four different methods: Walkley Black Method, Tube Digestion Method, Loss on Ignition Method and Potassium Permanganate Method.

Walkley-Black (WB) method: This was determined as described by Nelson and Sommers (1996)

Tube Digestion Method (Modified Walkley Black Method): The tube digestion method, popularly known as modified Walkley-Black method was described by Nelson and Sommers (1996). The procedure is similar to the Walkley Black procedure except that external heat (150 ºC) was applied to the reaction mixture.

Loss on Ignition Method: A crucible of known mass was weighed and about 3g of soil was weighed and placed into the crucible. The weight of the crucible and soil was recorded as W1. The soil plus crucible was placed in a furnace for two hours at 350°C. After which the crucibles were allowed to cool in a desiccator, weighed and recorded as W2. The percentage of organic carbon was calculated using the following relationship:

$$\% \text{ OM} = \frac{W1-W2}{W1} \times 100 \text{ - - - - - - - - - - - equation 1}$$

Based on the assumption that organic matter contains about 58% organic carbon, a correction factor of 1.72 was used to get the percentage of OC using the following relationship:

$\%OC = \frac{\% OM}{1.72}$ - - - - - - - - - - - equation 2

Potassium Permanganate Method

The adopted procedure for the determination of active or permanganate oxidizable carbon (POXC) as described by Weil *et al.* (2003). About 2.5 g of the air dried soil sample was weighed into a centrifuge tube. Two (2) ml of 0.2*M* potassium permanganate solution was added followed by 18ml of distilled water. The mixture was shaken for about 2 minutes on a mechanical shaker. After settling for 10 minutes, 0.5ml of the supernatant was transferred into another centrifuge tube followed by 49.5 ml of distilled water. 0.005*M*, 0.01*M*, 0.015*M* and 0.02*M* standard solutions were prepared by diluting 0.25ml, 0.5ml, 0.75ml and 1ml of permanganate stock solution respectively in a 10ml of distilled water. The absorbance of the sample and standards were read in a spectrophotometer at 550nm. The concentration of POXC was calculated using the following formula.

Permanganate Oxidizable Carbon POXC (mg kg^{-1}) =

0.02 mol/L - (a + b × Abs)] × (9000 mg C/mol) × (0.02 L solution/Wt) - - - - - - equation 3

Where: 0.02 mol/L = initial solution concentration
a = intercept of the standard curve
b = slope of the standard curve
Abs = absorbance of unknown
9000 = milligrams of carbon oxidized by 1 mole of MnO_4 changing from Mn^{7+}- Mn^{4+}
0.02 L = volume of stock solution reacted
Wt = weight of air-dried soil sample in kg
The value obtained was converted to % by dividing with 100.

Recovery factor and correction factor

The percentage recovery (RC) was computed by expressing the soil organic carbon content obtained by a particular method as a percentage to the soil organic carbon value obtained by the standard method (Walkley Black method) The formula for estimating recovery percentage is given in the equation:

$\%R = \frac{results\ by\ Evaluated\ method}{results\ by\ reference\ method} \times 100$ - - - - - - - - - - equation 4

The recovery factor (RF) for each method evaluated will be obtained using the average of percent recovery (%R) considering all samples evaluated by the equation (Bragança et al., 2015):

$RF = \frac{100}{\%R}$ - - - - - - - - - - equation 4

Statistical Analysis

The results of all methods were expressed in percentage and the data obtained were subjected to regression analysis using JMP statistical software.

Results and Discussion

Results

Table 1 shows the carbon values obtained using four different methods of determination; potassium permanganate, loss on ignition (LOI), Walkley-Black and tube digestion methods. The highest amount of carbon was observed with the use of loss on ignition and the lowest when potassium permanganate was used as an oxidizing agent. The amount of organic carbon obtained using permanganate ranged between 0.01 - 1.15 with a mean value of 0.48gkg^{-1}. The recovery percentage of 50.64% was observed and 1.98% as correction factor. Loss on ignition ranged from 0.23 - 2.33 gkg^{-1}, had a mean value of 1.24gkg^{-1} with a recovery percentage of 132.06% and 0.76% as correction factor. Tube digestion was observed to range from 0.45 to 1.96 gkg^{-1}, mean value of 0.75gkg^{-1}and having a recovery percentage and correction factor of 79.724% and 1.254%, respectively. The results showed that Walkley-Black which was used as the reference method range from 0.30 to 1.66 gkg^{-1}, with a mean value of 0.94gkg^{-1}.

The results of the regression analysis is as shown in Table 2. The observed relationship was poor as shown by the R^2 value obtained (< 0.5).

Table 1: Carbon values obtained by the different methods of soil organic carbon analysis

Method	Minimum	Maximum	Mean	Standard deviation	RC (%)	CF (%)
Potassium permanganate	0.01	1.16	0.48	0.27	50.64	1.98
Loss on ignition	0.23	2.33	1.24	0.47	132.06	0.76
Tube digestion	0.45	1.96	0.75	0.32	79.72	1.25
Walkley Black	0.30	1.67	0.94	0.47	-	-

RC – Percentage recovery; CF – Correction Factor

Table 2: Regression analysis between Walkley black and the other methods of organic carbon analysis

Methods		**Parameters of the linear regression**		
X	**Y**	**a**	**b**	**R^2**
Walkley-Black	Potassium permanganate	0.3003	0.1876	0.111
	Loss on ignition	1.134	0.1172	0.014
	Tube digestion	0.5327	0.232	0.113

a – Slope; b – Intercept; R^2 – Regression coefficient

Discussion

The highest values of soil organic carbon and recovery percentage were obtained when loss on ignition was used. The high results obtained may be attributed to the over estimation of the organic component of the sample as a result of the weight loss resulting from; the volatilization of hygroscopic and structural water along with the weight of organic compound lost in the form as CO_2 as explained by Ramamoorthi and Meena (2018) as well as Tunsisa and Culman (2016). Audette *et al.* (2016) and Mccarty *et al.*(2010) reported that that loss on ignition may overestimate the soil organic carbon.

The tube digestion method also showed a promising result when compared with potassium permanganate method(Roper et al., 2019)(Roper et al., 2019)(Roper et al., 2019). Abraham (2013) also reported a high value with the use of tube digestion method when compared with Walkley-Black, tube digestion and dry combustion. The result obtained was justified by the external application of heat under digestion tube hence resulting in the effective recuperation of total soil organic carbon in relation to the reference method. Since it oxidized more carbon, it can therefore be available alternative method for soil organic carbon determination.

The result was poorly related with the Walkley-Black but, tube digestion method have higher coefficient of regression value compared to the results of potassium permanganate and loss on ignition methods. The method that had lowest coefficient of linear regression with the reference method was the loss on ignition and can be attributed to the large variation in weight loss. In soils, the mineral fraction is greater than the organic one and part of the mineral components are hygroscopic or contain structural water. This is similar to the work of Ramamoorthi and Meena (2018). It is therefore, likely that these soil components loss water at high temperature.

High variations was observed in the percent recoveries of the different method which may probably be as a result of the incomplete oxidation of the organic carbon or due to the protection of the mineral fraction of the carbon (Roper et al., 2019). Amongst the four methods evaluated, LOI and tube digestion were observed to have the closest relation to the standard method which could be an indication of the efficiency of the method in organic carbon recuperation when compared with the use of permanganate (Ramamoorthi and Meena, 2018)

Conclusion

From the use of the four different methods investigated in this study, it can be seen that the use of Loss on ignition and tube digestion gave almost similar results to what was obtained with the conventional Walkley-Black method on the soils of Dakace and Soba of Northern Guinea Savanna of Nigeria. These methods may be used as an alternative to Walkley-Black in soil testing laboratories for soils of the Northern Guinea Savanna of Nigeria.

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