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Investigation note

Comparison of analytical methods for the determination of organic matter in soil with coffee waste

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Abstract

Organic matter is an indicator of soil quality and fertility and directly influences its physical, chemical, and biological properties. Its analysis is fundamental for agricultural and forestry production. The Walkley-Black method is the standard established in Mexican regulations; nevertheless, it has environmental disadvantages as it uses toxic chemical reagents with handling characteristics. For this reason, the calcination method is proposed as an alternative, which directly quantifies the content of organic matter in the soil by determining the weight loss of a sample when subjected to high temperatures. In this study, both methods were compared in soil samples enriched with solid coffee waste and with compost made from scw/bovine manure. The data were statistically analyzed to assess equivalence between the methods. The results showed a high correlation (r= 0.951) and absence of significant differences (p = 0.94), which supports the feasibility of using the calcination method as an alternative to the Walkley-Black method, given the high correlation in the values of organic matter when analyzed by both analytical methods. The possibility of modifying the current regulations to incorporate the calcination method is suggested, providing a safe and ecological option for determining organic matter in the soil.

Keywords:

calcination, fertility, Mexican regulations, soil analysis.

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Introduction

Organic matter (OM) is an essential component in soil; it is derived from the residues of plant and animal origin at various stages of decomposition, microbial biomass and complex organic substances, such as fulvic acids, humic acids and humins (Eyherabide *et al*., 2014).

It is considered one of the main indicators of soil quality and fertility, as it directly influences its physical, chemical, and biological properties (Ruiz *et al*., 2023). The determination of OM is essential to understanding the agricultural and forestry productivity of soils.

The most widely used method to measure this parameter is the one proposed by Walkley and Black (1934), known as Walkley-Black (WB), which estimates the content of organic carbon (OC) present in the soil; this analysis consisted of the oxidation of the sample with potassium dichromate in sulfuric acid. The reaction takes the heat from the acid solution, which raises the temperature and achieves the oxidation of organic carbon (Eyherabide *et al*., 2014).

To convert OC measurements into OM estimates, the Van Bemmelen factor is used, with a value of 1.724 (Pribyl, 2010; Bautista and Hernández, 2021). Although it has been used universally for many years, it is mentioned that the value can be influenced by factors such as vegetation cover, organic matter composition, profile depth, amount of organic matter and clay in the soil, as well as the degree of decomposition, which may reflect real differences in the carbon content of organic matter (La Manna *et al*., 2007).

The Walkley-Black method has the disadvantage of using large amounts of chemical reagents, such as potassium dichromate ($K_2Cr_2O_7$). Due to its high toxicity, this compound is classified as carcinogenic and can enter the body by inhalation or contact with the skin. Once inside, hexavalent chromium ions (Cr^{6+}) can cross cell membranes and be mistaken for sulfate anions (SO_4^{2-}), allowing them to reach the nucleus and react with DNA, causing mutations and genetic damage (DesMarais and Costa, 2019).

The application of this method generates this hazardous waste in large quantities, and, in many cases, laboratories lack adequate treatment systems, which can lead to the release of this waste into bodies of water, affecting both the environment and human health (Arévalo *et al*., 2022). The disadvantages of the WB method have prompted the search for other methods for soil OM estimation.

An alternative approach that has been proposed is the calcination method, which directly quantifies the OM content, since it is based on determining the weight loss of a dry soil sample by subjecting it to high temperatures (300 to 600 °C) in a muffle furnace for several hours (2-6 h) (Abella and Zimmer, 2007). This method is simple, does not cause damage to the environment, and does not require the use of hazardous reagents.

The Mexican standard, Official Mexican Standard NOM-021-RECNAT-2000, indicates that the WB method must be used for OM. Considering the aforementioned disadvantages and to provide evidence for a possible modification to the Mexican standard, this study used two analytical techniques to evaluate OM in samples of soil enriched with solid coffee waste (scw). In addition, mixtures of soil with compost, made from scw/bovine manure (bm) 75/25 v/v, were evaluated. Their differences were analyzed using a descriptive statistical test and linear regression.

Materials and methods

The experimental work was conducted in the laboratories of the Interdisciplinary Research Center for Regional Integral Development of the National Polytechnic Institute, Durango Unit (CIIDIR-Dgo, by its Spanish acronym).

Soil samples were collected in three localities of the state of Durango, Mexico: soil 1 (S1) in the municipality of Durango, coordinates 24° 03’ 05.1” north latitude and 104° 36’ 37.8” west longitude; soil 2 (S2) in the municipality of Cuencamé, 24° 55’ 42.5” north latitude and 103° 48’ 25.4” west longitude and soil 3 (S3) in the municipality of Durango, 24° 01’ 50.9” north latitude and 104° 36’ 57.1” west longitude.

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Solid waste from coffee (*Coffea* spp.) was added to these soils. This material is the insoluble part of the ground coffee bean that does not dissolve in hot water and remains after preparing the drink, coffee. The scw was collected at a chain of coffee shops in the City of Durango, Mexico.

Different mixtures were prepared, all on a dry basis, v/v. S1 was supplemented with 5, 10, 20 and 30% of scw; S2 with 5, 10 and 20%; S3 was supplemented with compost obtained from scw with bovine manure (bm), in percentages of 10 and 20%. Mixtures with 30% scw and 20% scw + 10% bm were also prepared with S3. All samples were ground in a laboratory mill. A total of 14 samples were analyzed in triplicate, both by the WB technique and by calcination.

For the analysis by the WB method, the sample was sieved through an 80-mesh sieve (0.18 mm). Zero point five grams were weighed and taken to a 500 ml Erlenmeyer flask. Ten milliliters of potassium dichromate (1N) were added, followed by 20 ml of concentrated sulfuric acid, and the mixture was left to stand for 30 min. After the time, 200 ml of distilled water, 5 ml of phosphoric acid and 5 drops of diphenylamine indicator were added. This process was also carried out with a control, which did not contain soil samples, only the reagents. The samples were then titrated with ferrous sulfate (1N) until a change from orange to dark blue was obtained.

To determine the percentage of organic matter, OC is first calculated using the following formula:

$$\%organicC = \frac{B - TgN0.39mcf}{g}$$

B= volume of ferrous sulfate spent to titrate the blank of reagents (ml); T= volume of ferrous sulfate spent to titrate the sample (ml); N= normality of ferrous sulfate; g= grams of sample; mcf= moisture correction factor.

The OM was calculated with the formula:

$$\%organicmatter = \%organicCx1.724$$

The calcination method involves the destruction of organic matter in the soil sample. The tests were performed with samples ground and sieved in an 80-mesh sieve (0.18 mm). First, the crucibles were brought to a constant weight using an oven at 105 °C for 24 h. Subsequently, the soil sample was added, and they were placed back in the oven at 105 °C for 24 h to remove moisture. They were then placed in the muffle furnace at a temperature of 550 °C for 5 h.

The OM content was calculated with the following formula:

$$\%OM = \frac{W1 - W2}{W1 - C} * 100$$

W1= dry soil weight; W2= soil weight after calcination; C= weight of the crucible.

For the statistical analysis, first, the normality of the data was verified using the Shapiro-Wilk test. Once normality was confirmed, Student's t-test was applied to assess significant differences between the methods. Subsequently, homoscedasticity was tested using Levene's test to verify the equality of variances between the compared groups. To evaluate the relationship between the two methods, Pearson's correlation coefficient was calculated and a linear regression analysis was performed to examine the relationship between the results obtained by both methods and to determine the possible equivalence between the measurements. All analyses were conducted in Microsoft Excel using the Real Statistics add-in.

Results and discussion

The results of the OM content in soils are shown in Figure 1. Regarding the addition of scw to the soil, S1 showed the best response in increase in OM, the addition of 5% increases three times the amount of OM compared to the initial value. In S2, the best response was obtained with the addition

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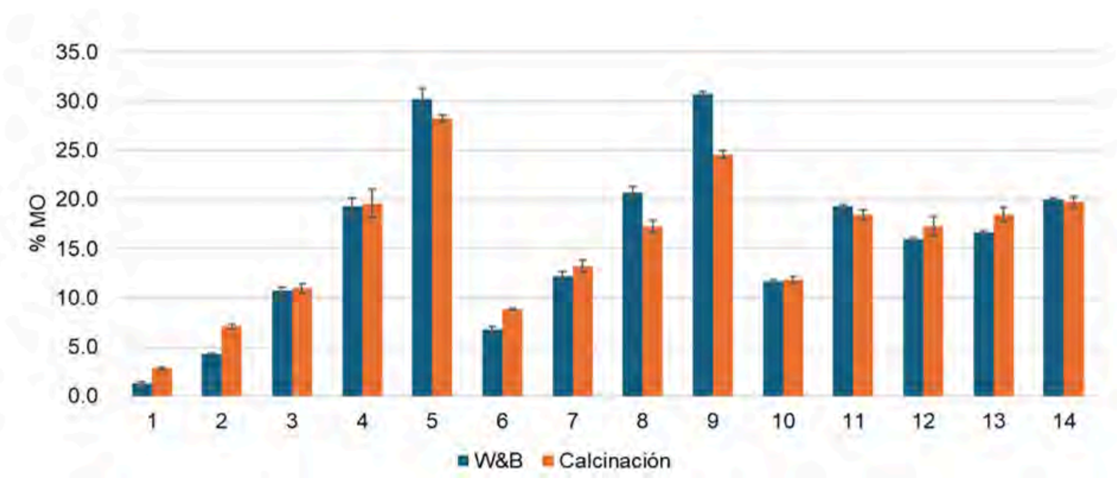
of 20% scw. In S3, which had the highest amount of initial OM, the addition of compost (20% and 10%), 30% scw directly (without composting) and 20% scw + 10% bm also directly, the increase in OM was similar in all treatments.

Figure 1. OM content in soils analyzed by WB and by calcination. 1(S1), 2(S1+5% scw), 3(S1+10% scw), 4(S1+20% scw), 5(S1+30%scw), 6(S2), 7(S2+5% scw), 8(S2+10% scw), 9(S2+20% scw), 10(S3), 11(S3+20% compost), 12(S3+10% compost), 13(S3+30% scw), 14(S3+20% scw+10% bm).



For the statistical analysis, first, the normality of the data was evaluated using the Shapiro-Wilk test, obtaining coefficients of 0.9487 and 0.9665 for the WB and calcination groups, respectively, with *p*-values of 0.0583 and 0.2508 ($p > 0.05$), which indicates that both sets follow a normal distribution, and this allows us to use parametric tests in the analysis.

Subsequently, the homogeneity of variances was verified by applying Levene's test, which did not show significant differences in the variances between the groups (means: $p = 0.2126$; medians: $p = 0.1989$; trimmed means: $p = 0.2095$), so homogeneity of variances is assumed, confirming the homoscedasticity between the groups.

With these conditions met, Student's t-test was used for related samples, obtaining a *p*-value of 0.6957, which suggests that there are no statistically significant differences between the two methods analyzed. In addition, Pearson's correlation analysis yielded a correlation coefficient of 0.9752 ($p < 0.001$), indicating a very high positive relationship between the results of both analytical methods.

Although the data exhibit some dispersion, the adjustment by linear regression shows a coefficient of determination (R^2) of 95%, indicating a strong relationship between the variables analyzed (Figure 2). This result suggests that the calcination method is a viable alternative for the determination of OM instead of the WB chemical method.

Revista Mexicana de Ciencias Agrícolas
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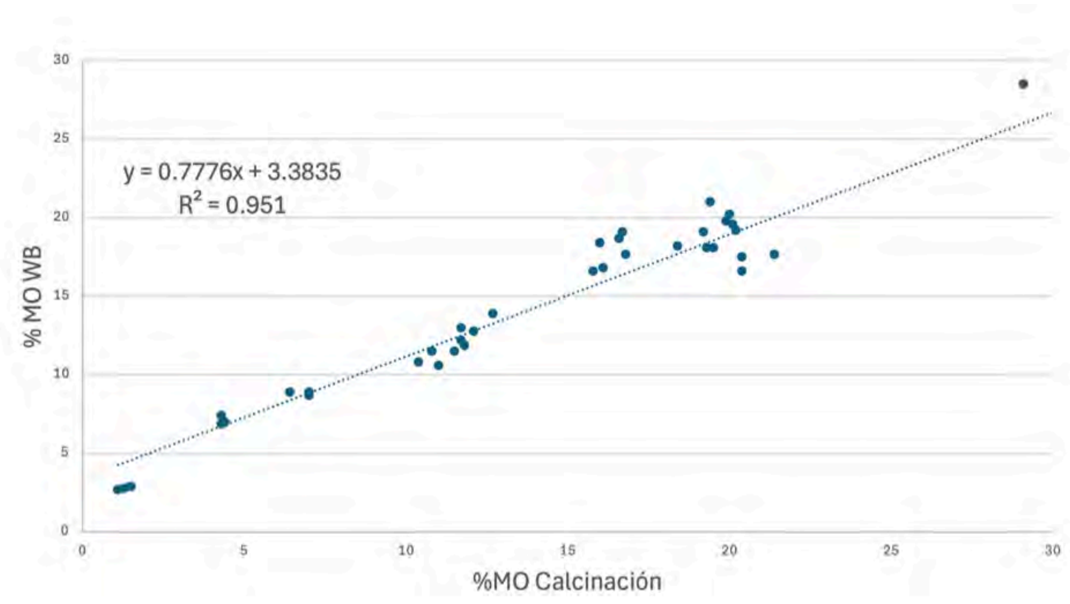
Figure 2. Linear relationship of OM in soils analyzed by WB and by calcination.



Several studies report on the comparability between WB and calcination methods for the determination of OM. A research paper by Ruiz *et al*. (2023) reported a correlation (R^2) of 0.90 after analyzing five organic materials in nine laboratories, without finding any significant differences.

In another study, Arévalo *et al*. (2022) analyzed soils with varying clay contents to determine the variability between methods, finding a correlation (R^2) ranging from 0.7 to 0.82, depending on the clay content. They mention that, regardless of this factor, there is a reliable relationship between both methods.

On the other hand, Eyherabide *et al*. (2014) report a regression of 0.98 in the analysis of soils with loam texture, without observing significant differences. In an analysis of 84 agricultural soil samples in North Carolina, Roper *et al*. (2019) found a strong correlation between WB, calcination, and dry combustion methods, concluding that either of these methods is equally efficient in determining soil OM content. These results together support the feasibility of the calcination method as a reliable and effective alternative to the WB method, regardless of soil classification.

Conclusions

There is a high correlation in the OM values when analyzed using two analytical methods -WB and calcination. The results obtained in the study, together with the evidence of previous research, support the possibility of suggesting a modification in the current NOM-021-RECNAT-2000 standard, to incorporate the calcination method as a standard option for the determination of OM in the soil. This method, in addition to being sustainable and environmentally friendly, is effective, safe and low-cost.

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Revista Mexicana de Ciencias Agrícolas
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