

Colorimetric method to estimate the soil organic matter in karst areas

Método colorimétrico para estimar la materia orgánica del suelo en áreas de karst

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ABSTRACT. The increase in soil organic matter (SOM) content contributes to the mitigation of the effects of global climate change; thus, it is important to know its levels. However, the SOM analysis can eventually be expensive and time consuming, as well as generating toxic waste. The measurement of soil color may be an indirect method more practical to estimate the SOM than traditional techniques. The principal aim of the study was to use color parameters through the CIE Lab system and some color indices, such as saturation and redness indices, to estimate the SOM in a karst area of the municipality of Chetumal in the Yucatan Peninsula, Mexico. The percentage of SOM was measured in 50 soil samples by conventional methods while the soil color was analyzed with the CIE Lab system. Both variables were correlated with the redness index. Based on color, the samples were separated into five groups, ranging from pinkish white to brownish gray. Multiple regression equations (SOM vs soil color parameters) were performed for each group and a medians comparison analysis was applied. The correlation adjustment between the redness index and SOM is $R^2 > 0.86$. The values of the multiple regression equations were $R^2 > 0.8$. We conclude that the soil redness index, now named soil organic matter index, can be used as a relatively quick approach to estimate the percentage of SOM in karst areas.

Key words: Organic matter, redness index, soil color, proxy method, karst.

RESUMEN. El aumento del contenido de materia orgánica del suelo (MOS) contribuye a mitigar los efectos del cambio climático global, por lo que es importante conocer sus niveles. Pero el análisis del MOS puede resultar costoso y tardado, además de generar desechos tóxicos. La medición del color del suelo puede ser un método indirecto más eficaz para estimar el MOS con respecto a los métodos tradicionales. El objetivo del estudio fue utilizar parámetros del color mediante el sistema CIE Lab y algunos índices de color, como los índices de saturación y de rojez, para estimar el MOS en una zona kárstica del municipio de Chetumal en la Península de Yucatán, México. El porcentaje de MOS se midió en 50 muestras de suelo mediante métodos convencionales, y el color del suelo se midió con el sistema CIE Lab. Ambas variables se correlacionaron con el índice de rojez. Según el color, las muestras se separaron en cinco grupos, que van del blanco rosado hasta el gris parduzco. En cada grupo se realizaron ecuaciones de regresión múltiple (MOS vs parámetros del color del suelo) y se aplicó un análisis de comparación de medianas. El ajuste de la correlación entre el índice de rojez y el MOS es de $R^2 > 0.86$. Los valores de las ecuaciones de regresión múltiple fueron de $R^2 > 0.8$. El índice de rojez del suelo, ahora nombrado índice de materia orgánica del suelo puede ser utilizado como una técnica rápida, para estimar el porcentaje de MOS en zonas kársticas.

Palabras clave: Materia orgánica, índice de rojez, color del suelo, método proxy, kárstico.

INTRODUCTION

The significance of soil organic matter has recently been recognized as a natural process of carbon storage that may help to mitigate climate change (Viscarra-Rossel *et al.* 2008, Powlson *et al.* 2011, Ontl and Schulte 2012). This has led to the organization of a worldwide network to develop large databases of soil organic carbon inventories (Paz and Etchevers 2016). However, the traditional method of analysis of organic matter in soils is relatively expensive, requires intensive laboratory work, and is definitively time-consuming polluting waste are produced from the analysis. A far more practical technique for determining a soil's composition is the indirect method of studying the soil's color for indications of its composition (Levin *et al.* 2005, Viscarra-Rossel *et al.* 2008, Cortés *et al.* 2015, Hausmann *et al.* 2016).

The soil color is the physical property of primary consideration in the identification of soil types (Spielvogel *et al.* 2004), soil ethnopedological classes (Bautista and Zink 2010, Sánchez-Hernández *et al.* 2018), and orders or primary groups of soils (IUSS Working Group WRB 2015). The study of soil color has also been widely used in the research of soil genesis (Kumaravel *et al.* 2010), as well as for the identification of fertile soils (Schulze *et al.* 1993, Leirana-Alcocer and Bautista 2014) and automated identification of soil horizons (Zhang and Hartemink 2019).

Some compounds that give color to the soil are minerals and organic matter. For example, the colors vary with the presence of iron oxides (Torrent *et al.* 1983, Schwertmann 1993, Levin *et al.* 2005, Viscarra *et al.* 2008), soluble salts such as calcium carbonate, gypsum and others (Sánchez *et al.* 2004), heavy metals (Cortés *et al.* 2015, Marín *et al.* 2018, Delgado *et al.* 2019) and organic carbon (Torrent *et al.* 1983, Bédidi *et al.* 1992, Schwertmann 1993, Lindbo *et al.* 1998, Viscarra-Rossel *et al.* 2008, Vodyanitskii and Savichev 2017).

Colorimeters for the analysis of solid samples, such as soil, have been manufactured in recent years. At the same time, several color systems have been

developed that can be expressed numerically, as CIE-RGB, CIE-L*a*b* and CIE-XYZ (Leirana-Alcocer and Bautista 2014, Cortés *et al.* 2015, Aguilar *et al.* 2013, Marín *et al.* 2018). These color measurement systems allow mathematical relationships to be established with other soil properties (Leirana-Alcocer and Bautista 2014, Levin *et al.* 2005, Cortés *et al.* 2015, Marín *et al.* 2018, Delgado *et al.* 2019). The CIE-L*a*b* parameters are useful in obtaining the optimum redness index (Kirillova *et al.* 2014), which is helpful in determining the presence and contribution of Fe-oxides on percentage (Vodyanitskii and Savichev 2017). L* represents the contrast ranging from black to white (0-100) and a* and b* are chromatic coordinates, a* being the variance from red to green and b* that from yellow to blue (CIE 1978).

Simon *et al.* (2020) concluded that the relation between the color of soil and other properties as organic matter, texture, soil chemical composition, and particle size are variables; thus it is necessary to develop precise predictive models under soil specific properties of each place. Shields *et al.* (1968), indicated that the concentration and nature of the organic carbon in the organic matter generate several colorations. According to Chen *et al.* (2018), the coordinates a y b of a system of colors CIE L*a*b*, correlate intimately with soil organic carbon concentration.

In the tropical karst areas of Mexico on the Yucatan peninsula, there are large areas with soils of contrasting colors, which vary between white limestone rock and black organic matter (Bautista *et al.* 2003, Bautista 2021, Fragoso *et al.* 2017). To improve the accuracy of soil organic carbon inventories, it will be necessary to analyze thousands of soil samples; for this reason, it will be essential to generate models for estimating soil organic matter with proxy technologies. Hypothetically, the physical and chemical characteristics of a karstic soil don't impede the development of robust models to predict the organic matter concentration from soil color properties. Thus, the aim of this study was to explore the use of soil color parameters in order to estimate the organic matter in soils from a karstic zone in the Yucatan Peninsula in México.

MATERIALS AND METHODS

The study zone is in Chetumal (Quintana Roo), the south Yucatan Peninsula at the southeastern part of Mexico. This region is a large limestone plain with a tropical climate, where the Leptosols dominate the poorly developed karst plains, although there are also other soil groups such as Gleysols, Vertisols, Phaeozems, and Luvisols (Bautista *et al.* 2011, Fragoso *et al.* 2017). On the peninsula of Yucatán, the dominant vegetation is the low and sub-deciduous tropical forest and medium and perennial tropical forest.

Fifty soil samples were collected; these samples were air-dried in the shade and sieved with a 2 mm mesh. The soil samples were selected from a set of samples considering that the colors should be between the white color where limestone predominates and the black color due to the high percentage of organic matter in the soil. The chemical analyzes performed on the soil samples were: pH (Lean 1982), organic matter was measured using the wet oxidation method with potassium dichromate (Nelson and Sommers 1982), exchangeable cations Ca, Mg, Na and K with ammonium acetate (Okalebo *et al.* 1993).

An X Ray Diffraction (XRD) analysis was performed to identify minerals present in a soil sample. As calcite was the dominant mineral and prevents the identification of residual minerals, 10% HCl was added to destroy the carbonates. A soil sample was placed on a silicon sample holder coated with silicone grease suitable for XRD; subsequently, they were analyzed on a Siemens D-5000 diffractometer, Bragg-Brentano Mode, with a monochromatic Cu tube ($\lambda = 1.5418 \text{ \AA}$), a step time of 3 seconds, step size 0.02 degrees, at 34KV and 25 mA.

The organic matter index

The color of the soil samples was analyzed using a Konica Minolta CR-5 reflectance and transmission colorimeter. The color parameters were obtained using the system CIE-L*a*b* and CIE-XYZ defined by the International Commission on Illumination (CIE). The use of the CIE-L*a*b* simplifies

and strengthens statistical calculations (Vodyanitskii and Savichev 2017) and the CIE-XYZ is the base of the transmission to any other color space (Viscarra-Rossel *et al.* 2006).

In the CIE-XYZ model, X is the color red, which varies from 0 to 0.9505, Y is the color green, which varies from 0 to 1.0, and Z is the color blue, which varies from 0 to 1.089 (Kirillova *et al.* 2014). The redness index was introduced by Barron and Torrent (1986) to estimate the percentage content of hematite in soils, but it should simply be called "color index" like this in general, because it includes all the parameters of the CIE-L*a*b* color system and therefore can be associated with the materials and minerals that give the soil its color (Bautista *et al.* 2003; 2005),

$$RI = L^*(a^{*2} + b^{*2})^{0.5} 10^{10} / b^* L^{*6}$$

where RI = redness index, L* = luminosity, a* = coordinates of red/green, and b* = coordinates of yellow/blue. In this paper we will refer to this as the organic matter index (OMI).

The relationship between the soil organic matter (SOM) and the OMI is established under the assumption of a possible curvilinear behavior (Schulze *et al.* 1993), and proposes an adjustment based on the power regression analysis with the two terms.

$$SOM = aOMI^b + c$$

where a, b and c are the coefficients that should be found with a confidence limit of 95%; however, another adjustment is proposed with a logarithmic regression analysis (Viscarra-Rossel *et al.* 2008).

$$SOC = aLn(OMI) + b$$

where a and b are the coefficients that should be found for both equations with a confidence limit of 95% using regression analysis. A cross validation between the SOM and the equations obtained (Eq. 2, Eq 3) was applied in order to verify the viability to obtain the percentage of SOM.

Formation of soil sample groups by color

The soil samples were separated into color groups using color parameters following the methodology established by Cortes *et al.* (2015). The first step was to transform the parameters of CIE-XYZ system to the RGB system to rescale the XYZ triplets and subsequently make use of the square matrix 3 x 3 for the standard illuminant D₆₅ at 2° (Viscarra-Rossel *et al.* 2006).

$$\begin{pmatrix} R \\ G \\ B \end{pmatrix} = \begin{bmatrix} 3.240579 & -1.537150 & -0.498535 \\ -0.969256 & 1.875992 & 0.041556 \\ 0.055648 & -0.204043 & 1.057311 \end{bmatrix} \begin{pmatrix} X \\ Y \\ Z \end{pmatrix}$$

The CIE-RGB was used to make the cluster analysis and separate the soil samples into groups based on the similarities and differences between the color parameters with a k-means clustering (Matlab 2020a).

Based on the study of Vodyanitskii and Savichev (2017), we use the brightness (L), redness (a) and yellowness (b) to make a linear regression analysis for each group obtained.

$$SOM_i = X_L L_i + X_a a_i + X_b b_i + X_0$$

The parameters of color form a rectangular matrix $MAT_{N,4}$, and the $SOM_{N,1}$ is a vector of the soil organic matter.

$$SOM_{N,1} = MAT_{N,4} X_{4,1}$$

The vector of the coefficients $X_{4,1}$ is determined by the method of least squares with the Moore-Penrose pseudo-inverse algorithm.

The present study used the measure of error referred to as the K-factor ($\Delta SOM / SOM^{1/2}$) (Vodyanitskii and Savichev 2017). The multiple determination coefficient R^2 was applied to observe whether the estimated SOM_e was accurate about the original SOM. The number of the samples should be quite large ($N > 8$) and the content of the SOM greater than 0.4% to obtain a strong correlation (Vodyanitskii and Savichev 2007).

Finally, the SOM and OMI by color group of soils were compared using the Kruskal-Wallis test, as

it is the best method to compare population in which there is no gaussian distribution of the data. Kruskal and Wallis test (1952) evaluates the hypothesis that the median of each group is equal; it combines the data of every group and orders them from least to greatest, and subsequently calculates the average range for the data of each group.

RESULTS

The organic matter index

Descriptive statistics provided the values of the variation of the SOM and the OMI (Table 1) in all of the samples, making it possible to observe the variation of the SOM in its concentrate between 2.11 ± 1.30 and that of the OMI between 18.80 ± 18.28 with a median similar to the mean that indicate that the samples belong to the same group of soil samples. Other chemical properties of soils also show a wide range of variance, such as the CEC ranging from 18.9 to 34.7 cmol kg^{-1} (Table 1).

The SOM vs. OMI has two mathematical adjustment by power and logarithmic fit with a small deviation that ensured the correlation between both parameters with an $R^2 > 0.86$ and a $R^2 > 0.87$ (Table 2, Figure 1) respectively. The relation that exists between SOM vs SOMI provided an equation to estimate the soil organic matter (SOM_e) and when is made a cross-validation between the SOM (measured) and the SOM_e was obtained a $R^2 > 0.85$ and the RMSE 0.50% which indicates a clear association.

Formation of soil sample groups by color

The full set of samples was organized into five groups divided by color: Group I with soils of a pinkish white color, Group II with soils of a brownish grey color, Group III with soils of a grey color, Group IV with soils of a greyish brown color, and Group V with soils of a dark grey color (Table 3, Figure 2). These soil colors have significative difference for the SOM and OMI observed in Figure 3 by the box plot.

These five groups were independently related with their SOM by a multiple linear system. The correlation of each of the groups is well defined, especially for Group V, where the system that it conformed

Table 1. Chemical properties of soils.

| | pH | EC | Ca | Mg | K | Na | CEC | SOM | SOMI |
|-----|-----|------|---------|-----|-----|-----|------|-----|------|
| | | | cmol/kg | | | | | % | |
| X | 7.8 | 0.59 | 36.1 | 2.6 | 0.2 | 0.4 | 25.2 | 2.1 | 18.8 |
| Sd | 0.2 | 0.13 | 6.5 | 0.7 | 0.1 | 0.4 | 4.0 | 1.3 | 18.2 |
| Max | 8.3 | 1.19 | 48.9 | 4.7 | 0.4 | 1.2 | 34.7 | 5.1 | 92.4 |
| Min | 7.4 | 0.39 | 24.2 | 1.6 | 0.0 | 0.0 | 18.9 | 0.0 | 2.7 |

EC = electrical conductivity; CEC = cation exchange capacity; SOM = soil organic matter; SOMI = soil organic matter index.

Table 2. Regression analysis, equation for correlation and cross-validation.

| Regression analysis | R ² | SOM vs. SOM _e | Cross-validation (R ² , RMSE) |
|--|----------------|--------------------------|--|
| SOM = 9.75(SOMI) ^{0.101} - 10.5 | 0.86 | f(x) = 0.86x + 0.30 | 0.85, 0.51 |
| SOM = 1.26Ln(SOMI) - 1.05 | 0.87 | f(x) = 0.85x + 0.31 | 0.85, 0.52 |

R² = the square correlation between the response values and the predicted response values; SOM = Soil's Organic Matter; SOM_e = Estimated Soil Organic Matter; SOMI = organic carbon index RMSE = Root Mean Squared Error.

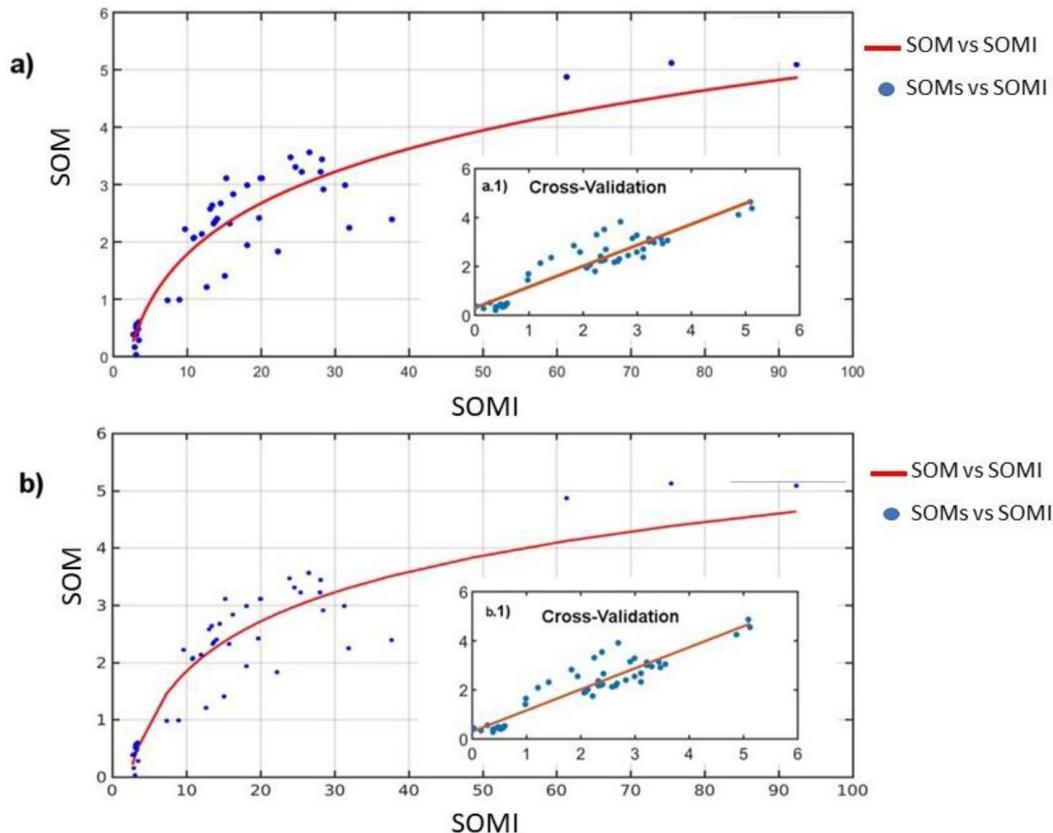


Figure 1. Fit analysis between the SOM and the SOMI: a) Power regression analysis and cross-validation, b) Logarithmic adjustment and cross-validation.

was quite outstanding by the mathematical analysis due to the matrix $SOM_{4 \times 4}$ being quadratic, which ensured the solution of the system. The amounts of the

SOM have the major percentage for the group V and gradually decrease until the group I (Table 4).

The multiple linear correlations between the

Table 3. Descriptive statistics of the soil organic carbon in the five color groups.

| Groups/Colors | I Pinkish white | II Brownish grey | III Grey | IV Greyish brown | V Dark grey |
|---------------------|-----------------|------------------|----------|------------------|-------------|
| Mean SOM (%) | 0.55 | 1.66 | 2.36 | 2.96 | 4.44 |
| Maximum | 1.21 | 2.22 | 3.11 | 3.56 | 5.12 |
| Minimum | 0.16 | 0.48 | 0.03 | 1.83 | 2.69 |
| Standard deviation. | 0.27 | 0.73 | 0.79 | 0.57 | 1.17 |
| N | 13 | 6 | 16 | 11 | 4 |

n = sample number.

| Groups | I | II | III | VI | V |
|--------------------|---|---|---|--|---|
| Samples and Color. |  |  |  |  |  |
| | Pinkish white | Brownish grey | Grey | Greyish brown | Dark grey |
| L* | 77.9 | 43.5 | 51.8 | 59.3 | 65.7 |
| a* | 4.1 | 2.3 | 2.4 | 2.4 | 2.6 |
| b* | 13.4 | 7.7 | 9.0 | 9.6 | 10.6 |

Figure 2. Color groups of soil samples. L* = luminosity, a* = coordinates of red/green, and b* = coordinates of yellow/blue (CIE 1978).

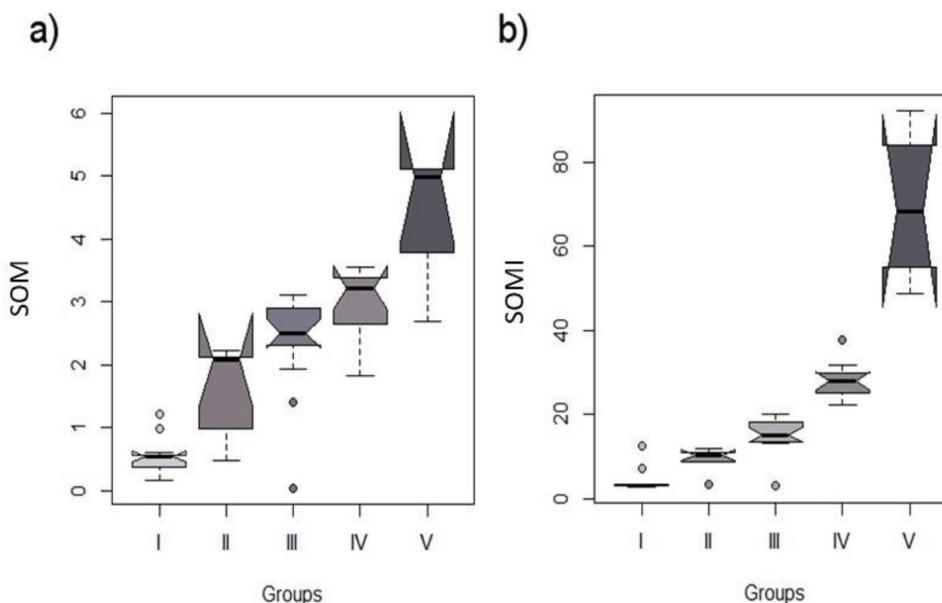


Figure 3. Box plot for the five groups: a) SOM, b) OMI.

CIE-L*a*b parameters and the SOM provided good results for each group, especially for Groups I, II, and V. This further emphasizes the relationship that

exists between the three parameters of color in the CIE-L*a*b* system with the SOM, providing equations to estimate the soils organic matter estimated in the

Table 4. Linear regression between the soil's organic carbon and parameters of color.

| Group | SOM (%) | SOM = $X_L L^* + X_a a^* + X_b b^* + X_0$ | SOM _e (%) | R ² |
|--------------------|--------------------------------------|---|--------------------------------------|----------------|
| I (pinkish white) | 0.55 ^{0.82} _{0.28} | 3.41 - 0.043L* - 0.16a* + 0.084b* | 0.54 ^{0.80} _{0.28} | 0.92 |
| II (brownish grey) | 1.66 ^{2.39} _{0.93} | 12.96 - 0.15L* + 0.83a* - 0.34b* | 1.66 ^{2.31} _{1.01} | 0.89 |
| III (grey) | 2.36 ^{3.15} _{1.57} | 5.75 + 0.01L* - 0.14a* - 0.36b* | 2.56 ^{3.20} _{1.92} | 0.85 |
| IV (greyish brown) | 2.96 ^{3.33} _{2.39} | 12.18 - 0.24L* - 6.67a* + 2.14b* | 2.79 ^{3.26} _{2.32} | 0.83 |
| V (dark grey) | 4.44 ^{5.61} _{3.27} | 50.53 - 0.75L* - 16.7a* + 3.33b* | 4.47 ^{5.65} _{3.30} | 1.0 |

SOM = the soils' organic matter, SOM_e = soils' organic matter estimated by Group, R² = the square coefficient of correlation.

Groups (SOM_{eg}).

Groups II and V did not meet the first requirements of the number of samples N > 8 for a suitable adjustment, but despite this, the results present a substantial correspondence.

DISCUSSION

The organic matter index

The value of the correlation between SOM (measured) and SOM_e (estimated) in this study was acceptable compared to that obtained by Stiglitz *et al.* (2017). They used the soil color parameters as a predictor of the SOM developing a prediction model, using the soil depth, L* and a* as independent variables in dry soils obtaining values of R² = 0.7978 and RMSE = 0.0819. In contrast, in soils wet R² = 0.7254 and RMSE = 0.1536. These results suggest that the soil color is efficient for the rapid determination of SOM. However, they warn that the high iron contents, carbonates, depth, and the humidity of the soil are variables that can negatively affect the model's predictive capacity. To improve the accuracy of color measurement with electronic equipment we recommended taking into account: a) the size of the particle (Sánchez *et al.* 2004); b) soil moisture (Domínguez *et al.* 2012); c) particulate organic matter.

A better fit was obtained between SOM and SOM_e because the parameter L (luminosity) plays an important role due to the colors of the soil samples vary from white (Calcite) to black (humidified organic matter) (Figure 2).

The content of exchangeable cations (Ca, Mg, Na, and K), the pH value, electrical conductivity, and the cation exchange capacity are typical of soils de-

veloped on limestone (Bautista *et al.* 2011).

Calcite and, to a lesser extent, quartz are the minerals that appear in the diffractograms. Once the Calcite is eliminated with HCl, other minerals appear, such as Tosudite (white, light yellow, light green), Hematite (red), Dickite (white), Boehmite (white), and Goethite (brown) (Figure 4); in addition, the sample turns darker in color because both the Calcite and the Quartz are white, which gives the soil sample more luminosity. As occurs in Leptosols of karst origin of the Yucatan peninsula (Bautista *et al.* 2011). The color of the mineral fraction of the soil must be taken into account because, in some cases, it is the one that dominates the color of the soil, mainly iron minerals (Barron y Torrent, 1986, Schwertmann 1993, Schulze *et al.* 1993).

Formation of soil sample groups by color

According to Simon *et al.* (2020), SOM has a great soil darkening capacity, which even masks the white colors of minerals. This property can be adequately predicted through color due to the strong relationship between the color and nature of the soil organic matter. Humic acids with higher carbon richness had darker colors (Shields *et al.* 1968), which explains the dark gray color of group V and the higher percentage of organic matter.

In the opinion of Chen *et al.* (2018), the CIE L* a* b* color system alone can predict the percentage of SOM, although multiple linear regression analysis can marginally improve the prediction; the b* coordinate correlates negatively with the concentration of SOM, mainly expresses the yellowish colors, so it is related to the low concentration of SOM; the a* coordinate, on the other hand, exhibits a stronger correlation with brownish colors, while the L* coordi-

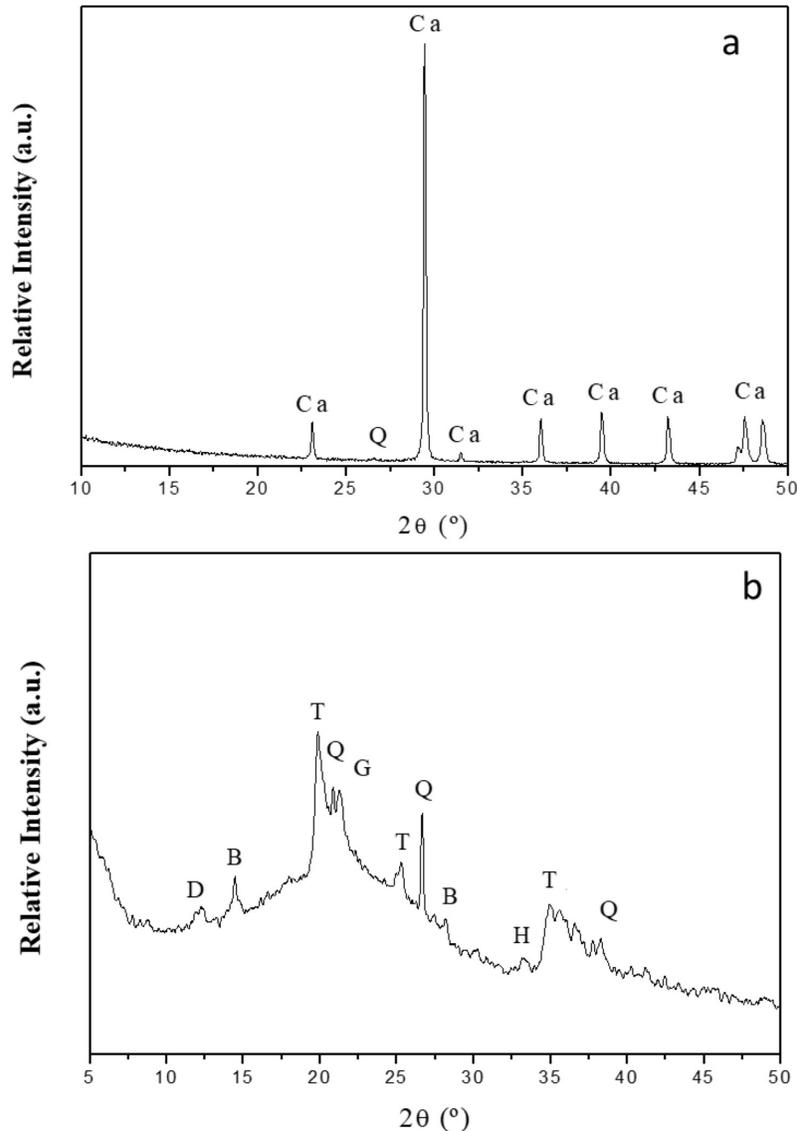


Figure 4. Soil minerals with (a) and without calcite (b). Ca = calcite, Q = Quartz, T = Tosudite, H = Hematite, D = Dickite, B = Boehmite, G = Goethite.

nate shows a low correlation with the concentration of the SOM.

The five equations obtained (Table 4) for each group can be used to estimate organic matter in large collections of soil samples in karst areas; however, further equations must be generated for soil samples with other colors such as reds, yellows and browns that also exist in karstic zones from peninsula of Yucatan (Bautista *et al.* 2003, Bautista *et al.* 2005, Bautista *et al.* 2011).

The association between soil color and organic matter is widely known (Schulze *et al.* 1993); the mathematical model proposed between the color components (L^*a^*b) and the percentage of organic matter is relevant in this study. In this same sense, other mathematical models have been proposed (Spielvogel *et al.* 2004, Domínguez *et al.* 2012, Stiglitz *et al.* 2017, Chen *et al.* 2018); however, they are very different because the soils are also different in mineralogy and the type of organic matter and par-

title size.

CONCLUSIONS

The SOMI allowed to estimation the soil's organic matter using both Power and Logarithmic fit. The grouping of soil samples by color allowed to describe a linear relationship between the soil color and its organic matter percentage, which improved the efficiency of this proxy technique. The darker or lighter colors as dark grey and pinkish-white, showed a higher level of R^2 , concerning other colors as brownish grey, grey, and greyish brown. Thus, the correlation sequence of color groups is V (dark grey) > I (pinkish white) > II (brownish-grey) > III (grey) > IV (greyish brown). For the karstic conditions of the Yucatan Peninsula, the study of soil color (SOMI and color parameters) may be considered useful for the estimation of the organic matter in large collections of soil samples, even in samples with low SOM content. Furthermore, this technique is much cheaper and less time-consuming compared to the traditional

methodology because of its straightforward treatment allowing fast measurements. Another notable benefit of this technique for estimating soil organic matter is that, unlike the conventional method, it produces no environmentally harmful waste products.

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