

**Division - Soil in Space and Time** | Commission - Soil Survey and Classification

# Histosols in an Upper Montane Environment in the Itatiaia Plateau

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ABSTRACT: Highland environments favor accumulation and preservation of soil organic matter (SOM) due to low temperatures, leading to the formation of Histosols. The Itatiaia National Park (INP), Rio de Janeiro, Brazil, offers conditions for preservation of SOM deposited over time, which has led to the formation of these soils. The objective of this study was to characterize Histosols in this environment, with the premise that it may provide evidence of changes in vegetation. Organossolos (Histosols) were sampled, characterized, described, and analyzed for their properties, stable isotopes of <sup>12</sup>C and <sup>13</sup>C, <sup>14</sup>N and <sup>15</sup>N, and dating of organic matter through <sup>14</sup>C. The soils were classified in the Brazilian Soil Classification System as Organossolo Háplico Hêmico típico - RJ-01 (Haplohemists) and Organossolo Fólico Sáprico cambissólico - RJ-02 (Udifolists). The morphological properties, degree of transformation, and chemical fractioning of SOM were consistent with hemic and sapric materials. The  $\delta^{13}$ C and  $\delta^{15}$ N isotopic analyses showed a difference in the contribution of plant materials. In RJ-01, there was previous influence of algae, due to poor drainage, and depletion of  $\delta^{13}$ C, suggesting a mix of C<sub>3</sub> and C<sub>4</sub> plants. RJ-02 showed influence of C<sub>3</sub> type plants. The <sup>14</sup>C dating for RJ-01 was  $3280\pm80$  years, and for RJ-02,  $2005\pm5$  years (modern age).

**Keywords:** peat lands, *Itatiaia* National Park, environmental changes.

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#### INTRODUCTION

Histosols in a tropical climate are formed at two different altitudinal zones. The first, which is more common, is at elevations lower than 30 m, called low altitude peat lands (lowland environment). The second, which are at elevations above 1,200 m, are called mountain peat lands (upper montane environment) (Chimner and Karberg, 2008; Benavides and Vitt, 2014). Both of these are found in South America, Africa, and Papua New Guinea (Page et al., 2011).

The *Itatiaia* plateau is located in the Mantiqueira Ridge, exhibiting an unique lithology, with mountainous terrain and rocky escarpments ranging from 2,000 to 2,791 m elevation, with its highest altitude in the Agulhas Negras Peak (Barreto et al., 2013). As a result of elevation, the weather is cold and wet, with endemic vegetation, characterizing the Histosol formations in the Itatiaia environment as upper montane (Benites et al., 2007; Chimner and Karberg, 2008).

In general, poorly developed soils are found in these environments, such as *Neossolos Litólicos* and *Cambissolos*, and rocky outcrops. However, Histosols can be formed (Simas et al., 2005; Benites et al., 2007; Chimner and Karberg, 2008; Scheer et al., 2011) by favoring the contribution of organic matter in detriment to its transformation, thus leading to its accumulation (Silva et al., 2013; Weissert and Disney, 2013; Bispo et al., 2015). In addition, they form as a result of a decrease in decomposition rates at low temperatures, while a constant supply of organic matter is maintained (Benites et al., 2007; Silva et al., 2009; Weissert and Disney, 2013; Benavides and Vitt, 2014; Cooper et al., 2015; Hribljan et al., 2016;).

These soils have great environmental/ecological importance, owing to their high carbon storage capacity, ability to recharge aquifers, use as a substrate for vegetation (including species that have adapted to this environment), and their capacity to buffer toxic elements (Weissert and Disney, 2013; Cooper et al., 2015).

Soil organic matter (SOM) can provide evidence to assess environmental changes (Benites et al, 2007; Cooper et al, 2015). It is a record of the origin, identity, and characteristics of past vegetation (Pessenda et al., 2004), since plants take up C differently according to their photosynthetic cycle (C<sub>3</sub>, C<sub>4</sub>, and CAM). From the natural abundance of isotopes of C and N in the SOM, it is possible to make inferences regarding the originating plants by establishing natural succession models (Pessenda et al., 2004; Silva et al., 2013). The interpretation of these analyses (<sup>12</sup>C, <sup>13</sup>C, <sup>14</sup>N, and <sup>15</sup>N) reveals information about previous vegetation and, when temporally synchronized with <sup>14</sup>C lifetimes, may indicate possible local environmental changes (Francisquini et al., 2014; França et al., 2015).

The Itatiaia National Park (INP), Brazil's first national park, has great environmental importance as a conservation unit, which justifies studies on soil limitations and weaknesses. Moreover, Histosols in an upper montane environment have distinct geomorphic features (Benites et al., 2007; Scheer et al., 2011) compared to those in floodplain and coastal plain environments.

It is assumed that SOM deposited over time in the upper montane environment of the INP has been preserved during the formation of Histosols, so that a study of these soils may reveal vegetation changes between previous and current environments. In this context, our study aimed to characterize Histosols in upper montane environments in the Itatiaia Plateau through morphological and physical descriptions, chemical properties, isotopic analyses of carbon and nitrogen, and dating by <sup>14</sup>C.

#### **MATERIALS AND METHODS**

## Characterization of the area

The area is located in the Serra da Mantiqueira within the Itatiaia National Park, Rio de Janeiro, in a full conservation unit spreaded over an area of 225.54 km<sup>2</sup>. For management



purposes, the park was divided into two zones based on altitude. This study was undertaken in the zone at the higher altitude range, which is characterized by mountainous and rugged terrain, with altitudes ranging from 2,000 to 2,791 m, culminating in the Agulhas Negras Peak (Barreto et al., 2013). The geology in the upper part of the INP is represented by the Itatiaia Alkaline Massif rocks that are placed over the crystalline basement gneisses of the Serra da Mantiqueira. This formation is rare, and the alkaline massif reaches high altitudes. The following types of rocks can be found: gneisses, nepheline syenite, syenite quartz, alkaline granite, and magmatic breccias, in addition to colluvial and alluvial sediments (Barreto et al., 2013).

Climate in the region is classified as Cwa, which corresponds to a sub-tropical climate with hot and rainy summers and cold and dry winters, with average annual temperature of 16 °C and average annual rainfall of 2,300 mm (Köppen, 1948). In the upper part, at the Agulhas Negras peak, frost occurs, and temperatures can reach -10 °C in the months from June to August (Barreto et al., 2013). Vegetation in the plateau is described as a high altitude rocky outcrop complex (Benites et al. 2007), formed by a mosaic of vegetation types associated with the Atlantic Forest. Vegetation in the study site consists of herbaceous graminoid plants, with a predominance of Cyperaceae and Poaceae, arranged in clumps, and fewer incidences of other species.

## Soil sampling and analyses

The two profiles were collected at different locations in the landscape (Figure 1), covering an altitudinal range of 300 m and at a horizontal distance of about 3 km from each other; measurements were made with a GPS unit (Garmin GPSMAP 78). The RJ-01 profile was collected at 23 K 0533769 7524084 (WGS 84) at an altitude of 2,100 m in a depression zone at the bottom of closed valley, where the water table was high (hydromorphic environment with poor drainage). The RJ-02 profile was collected from the lower third of the slope in a moderately drained area located at 23 K 0530905 7525596 (WGS 84) at an altitude of 2,400 m. The profile descriptions, both general and morphological, were registered according to Santos et al. (2005), and the soil was identified according to the Brazilian System of Soil Classification - SiBCS (Santos et al., 2013).

Characterization of organic material was carried out according to the methods recommended for *Organossolos* (Santos et al., 2013). The following aspects were analyzed: von Post

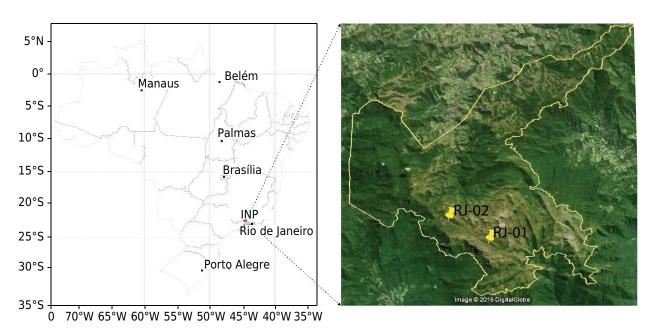


Figure 1. Profile locations within the landscape of Itatiaia National Park (INP), Rio de Janeiro, Brazil (RJ-01: 23 K 0533769 7524084 (WGS 84), elevation: 2100 m, and RJ-02: 23 K 0530905 7525596 (WGS 84), elevation: 2400 m).



humification scale, unrubbed fiber content (URF), rubbed fiber content (RF), pyrophosphate solubility index (PFI), organic matter (OM) and mineral material (MM) contents, minimum residue (MR), and the organic matter density (OMD). Physical and chemical properties were analyzed according to Silva (2009), and chemical fractionation of humic substances was carried out according to the method recommended by the International Humic Substances Society, with modifications suggested by Benites et al. (2003). Total C, H, and N were measured using the dry combustion method in the CHN elemental analyzer (TrueSpec Series: Carbon, Hydrogen, Nitrogen Elemental, Macro).

Soil samples were collected in trenches for isotopic analyses, from bottom to top in order to avoid contamination, at the following depths: 0.00-0.10, 0.10-0.20, 0.20-0.30, 0.30-0.40, 0.40-0.50, 0.50-0.60, 0.60-0.70, 0.70-0.80, 0.80-0.90, 0.90-1.00 m. An auger was used for collecting samples at the following depths: 1.00-1.10, 1.10-1.20, 1.20-1.30, 1.30-1.40, 1.40-1.50, 1.50-1.60, 1.60-1.70, 1.70-1.80, 1.80-1.90, and 1.90-2.00 m. Samples were ground, passed through a 100 mesh sieve, and then sent to the Stable Isotope Laboratory, in the Center for Nuclear Energy in Agriculture (*Centro de Energia Nuclear na Agricultura* – CENA) for determination of <sup>12</sup>C, <sup>13</sup>C, <sup>14</sup>N, and <sup>15</sup>N values. This was carried out in a Finnigan Delta Plus mass spectrometer coupled to a total C and N auto-analyzer, Carlo Erba AE1108 - Finnigan MAT, Bremen, having PDB as a standard for carbon and atmospheric air for nitrogen. All isotope collections were carefully conducted without contact with hands or other objects that could contaminate the samples with external carbon.

Soil samples were collected from a depth of 0.40-0.50 m for <sup>14</sup>C dating in the SOM. In order to obtain the humin fraction, roots and soil fauna were first manually cleared, following Gouveia et al. (1999). This was followed by removal of recent SOM, humic acid (HA), and fulvic acid (FA) fractions. This procedure is justified since, according to the author, dating all SOM fractions would underestimate the result. The HA and FA fractions are younger, and due to their higher solubility, they migrate from the surface, which has current residual input, to the subsurface, thereby masking the age of the original SOM that generated the *Organossolos*.

The humin fraction was sent to Stable Isotope Laboratory - CENA, where, for benzene synthesis, 70 g of humin was added to a tube containing copper oxide and lead chromate and heated at 650 °C for 60 min to form  $CO_2$ . This was collected and cooled with liquid nitrogen (-180 °C) and brought into contact with metallic lithium at 600 to 700 °C to form lithium carbide. At the end of the reaction, distilled water was added, causing hydrolysis and production of acetylene. Benzene was obtained by trimerization of acetylene in a catalytic converter at 90 °C for 2 h. Then, 0.5 mL of a scintillation solution (43.5 g of PPO and 2.59 g POPOP per liter of toluene) was added to the synthesized benzene, and the solution was kept in an unlit environment for 4 h for elimination of the phosphorescence effect. The solution was counted in a liquid scintillation spectrometer, Packard Tri-Carb 1550, with a low level of background radiation, for a period of 3000 min, at 100 min intervals. Data were then evaluated using specific mathematical procedures, and conventional  $^{14}$ C lifetime was corrected for isotopic fractionation.

# **RESULTS AND DISCUSSION**

#### Morphological properties and SOM characterization (SiBCS)

The RJ-01 and RJ-02 profiles presented histic diagnostic horizons (H and O) of 0.57 m and 0.59 m combined thicknesses, respectively (Table 1). These horizons are superimposed on mineral horizons, with an H-Cg sequence in RJ-01 and O-Bi in RJ-02. Identification of the Cg horizon in the RJ-01 profile is consistent with the high water table, conditioned by its position at the bottom of the valley, with drainage being influenced by bedrock outcrops and steep slopes (Benites et al., 2007; Scheer et al., 2011; Cipriano-Silva et al.,



2014). The color of the histic horizons (H and O) was uniform, which was 10YR3/2 in all horizons in RJ-01, and N2/ in most of the RJ-02 profile, thus ranging from very dark grayish brown to black (Table 1). In the RJ-02 profile, the Bi and Bi $_2$ /BC horizons exhibited a lighter matrix color (N4/). The colors seen in the histic horizons satisfy the concept of the *Organossolo* (Histosol) class in the SiBCS (Santos et al., 2013).

The long-term use of agricultural peats leads to changes in soil physical and chemical properties, particularly in layers that are not normally saturated with water, regardless of the initial vegetation composition (Könönen et al., 2015). Since the INP is a protected area, anthropogenic pressure consists of leisure activities along the park trails, and access to other areas is limited. Therefore, differences in morphological properties are more closely related to drainage, which in RJ-02 leads to greater variation in wetting and drying, due to the higher topographic position and absence of hydromorphism. Both the RJ-01 and RJ-02 profiles have material of an organic nature characterizing histic horizons. However, distinctions in the degree of decomposition of organic material are noticeable, since RJ-01 has higher amounts of fiber throughout the profile. The structure of the H and O horizons is granular, varying in size, in which RJ-02 has small and medium aggregates (Table 1). In RJ-02, sub-angular block structures, and even angular ones, were observed in the subsurface horizon (Bi), indicating a greater degree of structural development compared to RJ-01, which has massive structure in the Cg horizons.

The morphological properties and their location in the landscape indicate that the organic horizons (H) in the RJ-01 profile were formed by the paludization process ( $H_1$ ,  $H_2$ , and  $H_3$ ) and are followed by two horizons with evidence of gleization ( $Cg_1$  and  $Cg_2$ ). In RJ-02, organic horizons (O) are formed by the accumulation of organic matter ( $O_1$ ,  $O_2$ , and  $O_3$ ) favored by the low temperature, which reduces their transformation even under good drainage conditions, and the subsequent two horizons of a mineral nature have characteristics, such as variation in the block structure, which identify the subsurface horizon as B incipient (Santos et al., 2013).

**Table 1.** Description of morphological properties and degree of decomposition of organic matter indicators in *Organossolos* in the upper montane environment of Itatiaia National Park, Rio de Janeiro, Brazil

Duefile	Havinan	on Donth	Managhaslan	Tautum alasa	Comptumentia ii	vor	DE	DI	
Profile	Horizon	Depth	Munsell color	Texture class	Construction	Index	Material	RF	PI
		m						%	
			Organosso	lo Háplico Hêmico t	ípico (Haplohemists)				
RJ-01	$H_1$	0.00-0.12	10YR 3/2	Organic material	Mod, m/co, granular	$H_6$	Hemic	30	5
	H <sub>2</sub>	0.12-0.31	10YR 3/2	Organic material	Mod, m, granular	$H_6$	Hemic	27	4
	$H_3$	0.31-0.58	10YR 3/2	Organic material	Mod, m, angular blocky	$H_6$	Hemic	25	4
	$Cg_1$	0.58-0.76	10YR 3/2	Clay loam	Massive	$H_7$	Sapric	14	3
	$Cg_2$	0.76-1.0+	10YR 3/2	Clay loam	Massive	$H_8$	Sapric	15	3
			Organossol	o Fólico Sáprico car	mbissólico (Udifolists)				
RJ-02	$O_1$	0.00-0.15	N2/	Organic material	Mod, p/m, granular	$H_8$	Sapric	31	3
	$O_2$	0.15-0.28	N2	Organic material	Mod, f, granular	$H_8$	Sapric	18	3
	O <sub>3</sub>	0.28-0.59	N2	Organic material	Weak, f, subangular blocky	$H_9$	Sapric	17	3
	$Bw_1$	0.59-0,85	N2	Clay loam	Weak, f/m, angular blocky	$H_9$	Sapric	17	3
	Bw <sub>2</sub> /BC	0.85-1.0+	N4	Clay loam	Weak, f/m, angular blocky	$H_9$	Sapric	15	3

Level of development or grade (Mod = moderate); size (f = fine, m = medium, co = coarse);  $H_x$ : von Post scale values; RF: percentage of rubbed fibers; PI: sodium pyrophosphate index. According to Soil Survey Staff, the "w" suffix in RJ-02 designates weak color or structure within the B horizon; it is the suffix that corresponds to "i" in the SiBCS. For histic horizons, the nomenclature used in Brazil was preserved, separating H and O horizons according to the drainage formation environment.



The SOM transformation indicators in the histic horizons show different degrees of decomposition for profiles RJ-01 and RJ-02 (Table 1). According to the von Post scale, RJ-01 was reported as Hemic in the third taxonomic level due to the dominance of this degree of decomposition through most of the first meter of soil. In contrast, RJ-02 had a comparatively greater degree of transformation of organic matter throughout the profile, which identified it as Sapric. The values for the rubbed fibers index and the sodium pyrophosphate index (Table 1) complement the interpretation of the von Post scale in classification as *Organossolos* (Histosols) in the SiBCS. Better drainage in the RJ-02 profile favored mineralization and/or humidification by lowering fiber content, which explains the dominance of more processed materials (Sapric).

The two profiles are under similar climate and vegetation conditions at present, but show morphological differences in horizon sequences and in the degree of transformation of organic matter, as well as in the influence of topographical features. The hydromorphic environment, the presence of large amounts of fiber, and the degree of transformation in SOM in the histic horizon led to classification of the RJ-01 profile as *Organossolo Háplico Hémico típico* (Santos et al., 2013), corresponding to Haplohemists in Soil Taxonomy (Soil Survey Staff, 2014). In contrast, the RJ-02 profile, with better drainage and an incipient subsurface horizon environment was classified as *Organossolo Fólico Sáprico cambissólico* in the SiBCS (Santos et al., 2013), corresponding to Udifolists in Soil Taxonomy (Soil Survey Staff, 2014).

## **Chemical and physical properties**

The exchangeable complex of Histosols has characteristics different from other so-called mineral soils that act as vegetation regulators, causing the occurrence of a few species and promoting some species to emerge as dominants (Benavides and Vitt, 2014). The pH values ranged from 4.4 to 5.4, indicating medium acidity (Table 2). This is higher than values reported in other studies, such as 2.8 to 4.7 in the upper montane *Organossolos Háplicos* in Serra de Espinhaço, MG, Brazil (Silva et al., 2013) and 2.9 to 3.2 in Serra da Igreja, PR, Brazil (Scheer et al, 2011) and 2.7 to 3.8 for Histosols in Indonesia (Könönen et al., 2015). The higher pH values observed in the soils of the upper part of the INP could be a result of the alkaline parent material.

In general, the levels of exchangeable elements (Table 2) were low, with the exception of  $Al^{3+}$  and  $H^+$ , which are common in Histosols. Aluminum values were high in RJ-01 (ranging from 1.5 cmol<sub>c</sub> kg<sup>-1</sup> in the Cg<sub>1</sub> horizon to 6.0 cmol<sub>c</sub> kg<sup>-1</sup> in H<sub>1</sub>). However, in RJ-02 they ranged

Table 2. Chemical properties of Organossolos in the upper montane environment of Itatiaia National Park, Rio de Janeiro, Brazil

Profile	Horizon	pH(H <sub>2</sub> O)	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Al <sup>3+</sup>	H⁺	Na	K	Р	SB	Т	V
					cmol <sub>c</sub> kg <sup>-1</sup>	1		— mg	kg <sup>-1</sup> —	— cmo	l <sub>c</sub> kg <sup>-1</sup> —	%
	Organossolo Háplico Hêmico típico (Haplohemists)											
RJ-01	$H_1$	4.6	0.6	1.2	6.0	24.3	0.2	0.3	4.0	2.5	32.9	7
	$H_2$	4.6	0.5	1.1	4.3	25.2	0.2	0.1	1.0	2.1	31.7	6
	$H_3$	4.4	8.0	1.4	3.3	18.3	0.2	0.0	1.0	2.6	24.3	18
	Cg1	4.4	0.7	1.0	1.5	10.7	0.2	0.0	0.0	2.0	14.3	14
	Cg2	4.9	0.9	1.4	1.5	12.5	0.1	0.0	1.0	2.5	16.6	15
			Organos	solo Háplic	o Hêmico	o típico (Ha	plohemis	ts)				
RJ-02	$O_1$	5.0	0.3	0.9	1.7	11.5	0.3	0.2	1.0	1.8	15.1	11
	$O_2$	5.3	0.2	1.1	1.3	10.6	0.2	0.5	1.0	2.2	14.2	15
	O <sub>3</sub>	5.4	0.2	0.9	1.6	10.5	0.2	0.1	1.0	1.5	13.7	11
	$Bw_1$	5.1	0.2	0.9	1.5	7.1	0.2	0.1	1.0	1.5	10.2	15
	Bw <sub>2</sub> /BC	5.4	0.3	0.8	1.3	5.5	0.2	0.1	1.0	1.4	8.3	17

SB: sum of bases; T: cation exchange capacity; V: base saturation.



from 1.3 cmol $_c$  kg $^{-1}$  in Bw $_2$ /BC to 1.7 cmol $_c$  kg $^{-1}$  in O $_1$ . Hidrogen prevailed in the sorption complex of all profiles. However, the RJ-02 profile stood out as the lowest amplitude, 5.5 cmol $_c$  kg $^{-1}$  in the Bw $_2$ /BC horizon, up to 11.5 cmol $_c$  kg $^{-1}$  in the O $_1$  horizon. Due to the ability of SOM to complex Al, thereby reducing its toxicity in the soil solution, higher Al levels usually represent no limitation to plants in organic soils. In contrast, increased levels of hydrogen result from decomposition of plant residues wherein organic acids are released into the soil (Silva et al., 2009). The values of H $^+$  in the RJ-01 profile are related to hydromorphism, which leads to slow decomposition of plant material and permanence of acids that are distributed throughout the profile (Silva, 2009; Scheer et al., 2011).

The values of Ca, Mg, Na, K, and P were very low in both profiles, similar to Könönen et al. (2015) study, which found low values of nutrients other than P in Histosol without human intervention. In our study, Mg stood out as higher than Ca in all horizons, which could be due to the contribution of related rocks (nepheline-syenite). As a result of the low availability of nutrients, the sum of bases (S) ranged from 1.46 to 2.64 cmol<sub>c</sub> kg<sup>-1</sup>, and the cation exchange capacity (T value) from 8.36 to 32.93 cmol<sub>c</sub> kg<sup>-1</sup>. The very low base saturation (V) shows the influence of high values of H<sup>+</sup> in the SOM.

The physical properties used in *Organossolos* characterization and classification (Santos et al., 2013) are different from those used for soils with a predominance of mineral material. These properties are gravimetric moisture (% M), soil bulk density (Bd), particle density (Pd), organic matter density (OMD), minimum residue (MR), mineral material (MM), and total pore volume (TPV). They are related to organic carbon (OC) content, usually in a directly proportional manner. The values of these properties in the RJ-01 and RJ-02 profiles were similar (Table 3) and the organic matter content influenced all the physical variables.

The gravimetric moisture percentage was directly related to soil organic matter values, which is expected, given that it has large hygroscopicity (ability to capture and store water from the atmosphere). This is also a property commonly observed in Histosols, i.e., the large amount of water retained in organic horizons. The % M values decreased as depth increased, and in RJ-02, mineral horizons (Bw $_1$  and Bw $_2$ /BC) had the lowest values of % M. In contrast, MM values increased with depth and were inversely proportional to the organic matter content, with the highest value (98.22 %) in RJ-02 at Bw $_1$ . This pattern was also observed by D'Amore and Lynn (2002) and Silva et al. (2009).

**Table 3.** Soil bulk density (Bd), particle density (Pd), Organic matter density (OMD), minimum residue (MR), gravimetric moisture (Gm), mineral material (MM), total pore volume (TPV), and soil organic matter (SOM) of *Organossolos* of Itatiaia National Park, Rio de Janeiro, Brazil

Profile	Horizon	Bd	Pd	OMD	MR	Gm	MM	TPV	SOM
			— Mg m <sup>-3</sup> —		m m <sup>-1</sup>			%	
		Org	anossolo Há	plico Hêmico	<i>típico</i> (Hapl	lohemists)			
RJ-01	$H_1$	0.60	1.56	0.23	0.25	18.48	61.14	61.47	38.86
	H <sub>2</sub>	0.70	1.60	0.26	0.30	28.04	63.51	56.13	36.49
	$H_3$	0.70	1.72	0.19	0.34	14.42	73.00	59.63	27.00
	Cg1	0.74	2.00	0.13	0.41	11.98	82.42	63.10	17.58
	Cg2	0.82	1.92	0.18	0.43	12.61	78.27	57.15	21.73
		Org	anossolo Fóli	ico Sáprico ca	ambissólico	(Udifolists)			
RJ-02	$O_1$	0.54	1.55	0.24	0.20	45.14	55.73	65.30	44.27
	$O_2$	0.54	1.69	0.23	0.21	40.45	58.01	68.02	41.99
	O <sub>3</sub>	0.81	1.89	0.15	0.44	21.65	81.75	56.96	18.25
	$Bw_1$	0.95	1.98	0.10	0.56	8.93	89.22	52.13	10.78
	Bw <sub>2</sub> /BC	1.17	2.15	0.41	0.50	10.13	64.65	45.69	35.35



Both profiles showed similar TPV, with values decreasing with depth and directly related to organic matter content, ranging from 68 to 46 %. The higher values at the surface are due to recent addition of organic material, which has greater number of macropores; in the process of decomposition (or mineralization), these pores collapse, decreasing their size and affecting total porosity. TPV values were higher than those found by Scheer et al. (2011), who reported values from 10 to 33 % in upper montane Histosols at Serra da Igreja, Parana. However, the values were much smaller than those found by Könönen et al. (2015) in Indonesian Histosols.

In both profiles, Bd values increased with depth and were inversely proportional to SOM content. The highest Bd value occurred in RJ-02 in  $Bw_2/BC$  (1.17 Mg m<sup>-3</sup>), which is strongly influenced by the mineral fraction. Bd is a key physical property in Histosols, used in calculating OMD and as an indicator of the extent to which the organic matter is transformed (Lynn et al., 1947). Values are according to Silva et al. (2009), who observed variation from 0.03 to 0.44 Mg m<sup>-3</sup> in Histosols from Serra do Espinhaço, MG. Overall, the two profiles studied in *Itatiaia* showed decreasing OMD with depth. This is a direct reflection of a higher degree of humification and mineralization of organic matter, which in highland environments shows a distinct pattern with soil depth.

The MR values range from 0.20 to 0.56 m m<sup>-1</sup>, indicating the susceptibility of Histosols to degradation by the subsidence process (loss of volume). The MR is used to evaluate the maximum subsidence potential in horizons/layers formed of organic material, and are referred to as the thickness of the unit remaining after maximum subsidence (Lynn et al., 1974).

### Distribution of C, N, and carbon fractions in humic substances

Carbon values obtained by the CHN method were similar in their distribution across the soil depth in both profiles (Table 4). Moreover, in the surface horizons ( $H_1$ -RJ-01, and  $O_1$ -RJ-02) there is virtually no difference in C values (16.7 and 17.0 %). In these surface horizons, the cold weather and the upper montane environment have a similar influence on the type of vegetation (organism as the main soil formation factor). In the subsurface layers of the RJ-01 profile (with hydromorphism), an increase was observed in C values compared to the RJ-02 profile. Although these carbon values were low (Scheer et al., 2011, 2013; Könönen et al., 2015), according to Weissert and Disney (2013), these values are within the average found in peat soils, ranging from 8.4 to 47.3 %.

The N and H values followed the behavior for C, as they are both related to SOM. The amounts of C, N, and H determined by the dry combustion method were higher in surface horizons and decreased with depth. A similar distribution was reported in a study by Scheer et al. (2011) in a Serra do Mar (PR) upper montane environment.

The C/N ratio enables us to infer the dynamics of organic matter and its state of humification, and in Histosols, values are generally high (Broder et al., 2012). In RJ-02, values for the C/N ratio were high in the horizons of mineral formation (Bw<sub>1</sub> and Bw<sub>2</sub>/BC) and higher than those of organic horizons in the same profile. This result suggests higher SOM processing and, thus, interaction of organic and mineral material, contributing to greater stability of SOM. The C values in the chemical fractionation of SOM (Table 4) show high C content in the Humin fraction (C-HUM) in the surface horizons. This decreases with depth for both profiles, ranging from 165.24 g kg<sup>-1</sup> in H<sub>1</sub> to 27.12 g kg<sup>-1</sup> in Cg<sub>2</sub> in RJ-01, and from 159.43 g kg<sup>-1</sup> in O<sub>1</sub> to 15.71 in Bw<sub>2</sub>/BC in RJ-02. In all horizons, the C-HUM values were higher than those of other fractions. In general, C-HUM values are higher, as C-HUM includes C from remnants of leaves and stems, and even charcoal. The C-HUM values were higher in the RJ-01 profile, identified as Hemic. This confirms classification as *Organossolos* (Histosols), which was based on analysis of the property of scrubbed fibers and the von Post scale that highlights the difference in the degree of SOM humification.



**Table 4.** Carbon (C), hydrogen (H) and nitrogen (N) values, C/N ratio, C levels in the humic substances, and ratio of alkaline extract C-HAF/C-FAF in an upper montane environment in *Organossolos* of the Itatiaia National Park, RJ, Brazil

Profile	Horizon	С	Н	N	C/N	C-HUM	C-HAF	C-FAF	C-HAF/C-FAF
			— % —				— g kg <sup>-1</sup> —		
			Organ	ossolo Háp	lico Hêmico	típico (Haploh	emists)		
RJ-01	$H_1$	16.72	2.95	0.96	17.41	165.24	4.54	6.425	0.71
	H <sub>2</sub>	14.48	2.83	0.72	20.11	131.73	5.48	6.235	0.88
	$H_3$	10.01	2.39	0.47	21.29	70.68	5.93	6.575	0.90
	$Cg_1$	6.49	1.98	0.33	19.66	17.12	5.29	5.365	0.99
	$Cg_2$	7.39	2.11	0.40	18.47	41.91	5.86	5.575	1.05
			Organo	ossolo Fólic	o Sáprico ca	mbissólico (U	difolists)		
RJ-02	$O_1$	16.99	2.87	0.90	18.87	159.43	2.98	2.22	1.04
	$O_2$	10.56	2.32	0.60	17.60	64.42	3.59	2.65	1.36
	$O_3$	7.10	1.54	0.32	22.18	41.77	2.46	2.08	1.18
	$Bw_1$	4.56	1.16	0.18	25.33	22.75	1.47	1.21	1.22
	Bw <sub>2</sub> /BC	2.77	0.78	0.11	25.18	15.71	2.61	2.46	1.06

Carbon, hydrogen, and nitrogen obtained by the elemental analyzer method (CHN); C-HUM: C in the humin fraction; C-HAF: C in the humic acid fraction; C-FAF: C in the fulvic acid fraction.

A contrast was observed in carbon in the humic acid fraction (C-HAF), the values showed an increasing and erratic distribution with depth (Table 4), with RJ-01 ranging from 4.54 for H1 to 5.93 g kg<sup>-1</sup> for H<sub>3</sub>, and RJ-02 with lower values, ranging from 1.47 for Bw1 to 3.59 g kg<sup>-1</sup> for O<sub>2</sub>). RJ-01 had higher values of carbon in the humic acid (C-HAF) and fulvic acid (C-FAF) fractions in relation to RJ-02. As the former profile was from an area with a high water table, the removal of labile organic fractions from the soil section is prevented, leading to the accumulation of soluble fractions of organic matter throughout the length of the profile. Smaller amounts of C-FAF in RJ-02 suggest that SOM is still in an evolutionary stage, in the process of forming more stable structures. For the C-HAF/C-FAF ratio, there was variation from 0.71 to 1.05 in RJ-01, and 1.04 to 1.36 in RJ-02 (Table 4). These values reflect the degree of polymerization of the organic matter; the higher this value, the more condensed the organic matter will be. Thus, the ratio confirms the von Post levels, the fiber percentage, and the C-HUM content, which indicate a lower level of SOM evolution in RJ-01.

#### Isotopic composition of carbon and nitrogen, and dating of humin via 14C

The %C and %N values were similar between the profiles, as already mentioned, decreasing with depth (Figure 2). As soils are under the same type of vegetation and upper montane environment, such formative factors (climate and organisms) act with the same intensity as in current pedogenesis and, therefore, topography is a major factor in profile differentiation. C contents up to a depth of 0.20 m were equal for both profiles (Table 4), varying markedly at 0.30 m and with depth, with RJ-02 showing lower levels of C up to 1.80 m. This greater preservation of C in RJ-01 results from its poor drainage, favoring SOM accumulation and maintenance. N showed a similar response, while the C:N ratio varied widely in RJ-02, with values from 13 to 28, showing irregular distribution in the profile (Figure 2).

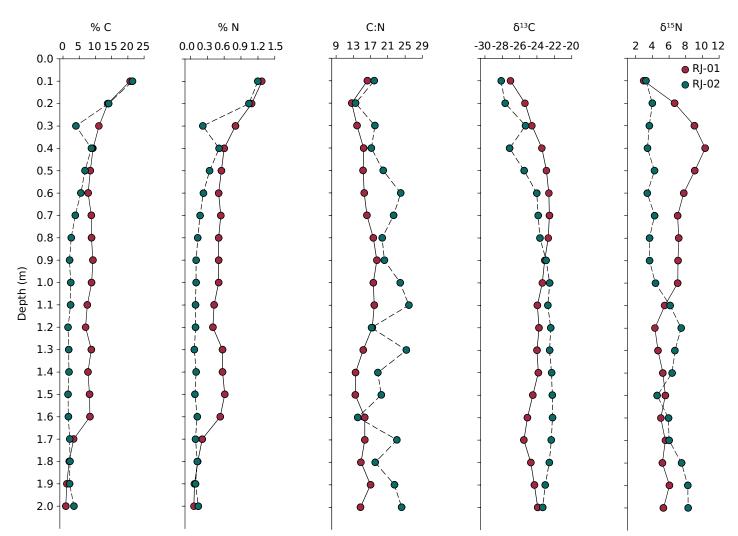
Histosols have an average C:N ratio of about 26, indicating balance between mineralization and immobilization processes (Cools et al., 2014). These values vary according to vegetation and edaphic and climatic conditions. The highest degree of transformation was observed in RJ-02, with C:N values of around 25 at various depths in the profile (0.60, 1.0, 1.3, 1.7, and 1.9 m). As this profile is characterized by better drainage, the organic material supplied tends to decompose faster, due to greater efficiency of aerobic



microorganisms in the SOM decomposition process. During the evolution of SOM, the values of C tend to stabilize, while those of N decrease. This is caused by the removal of N-rich groups such as amines, which increases the C:N ratio and results in a greater degree of humification (Silva et al., 2013.). For Histosols, this relationship is an indication of the degree of pedogenesis, due to the intense transformation of organic material that originates this soil.

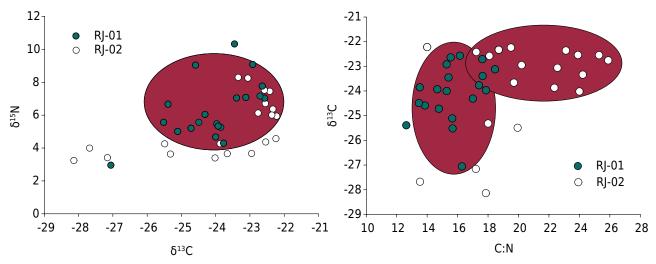
The  $\delta^{13}$ C values indicated a relative  $^{13}$ C depletion over time (Figure 2), particularly in RJ-02, with values ranging from -28 at present (0.00-0.10 m depth, surface horizon with recent supply of vegetation material) to -22 in the past (1.90-2.00 m depth, in the profile basis). According to this variation (a 6  $\delta$  shift), the vegetation in the profiles consisted of a mix of  $C_3$  and  $C_4$  plants, which shared the same environment for some time. Values close to -21 indicate a mix of vegetation types (Pessenda et al., 2004). Therefore, over the course of time, the proportion of  $C_3$  plants increased, leading to values of -28. The  $\delta^{15}$ N values ranged from 3 to 10, indicating changes in the local water system for both profiles. This was more pronounced for RJ-01, which exhibited a higher amplitude (Figure 2). Higher values indicate the preponderance of algal contribution to SOM (Peterson and Howarth, 1987), as seen at a depth of 0.30 to 0.50 m, which is possibly the average annual water table height in the RJ-01 profile.

Assessing the relationship between  $\delta^{13}C$  and  $\delta^{15}N$ , the values that fall within a range of 10 to 8 for  $\delta^{15}N$  and from -20 to -25 for  $\delta^{13}C$  (purple circle in Figure 3), correspond to



**Figure 2.** Distribution of carbon and nitrogen content, C:N ratio,  $\delta^{13}$ C and  $\delta^{15}$ N, for *Organossolos* in the upper montane environment of Itatiaia National Park, Rio de Janeiro, Brazil, with increasing depth.





**Figure 3.** Relationships between  $\delta^{13}C$  and  $\delta^{15}N$  (left) and between  $\delta^{13}C$  and C:N (right) for *Organossolos* in the upper montane environment of Itatiaia National Park, Rio de Janeiro, Brazil.

organic matter from algae, indicating a water saturated environment over long periods (Peterson and Howarth, 1987). As for the ratio between  $\delta^{13}C$  and C:N, two clusters can be seen (purple circle in Figure 3), where RJ-01 has greater proximity to organic material from algae, and the material supplied in RJ-02 shows a pattern similar to that originating from  $C_3$  plants (Peterson and Howarth, 1987). Thus, isotopic analysis of  $\delta^{13}C$  and  $\delta^{15}N$  allows the profiles to be differentiated according to the organic materials that contributed to the SOM, with earlier influence of algae in the RJ-01 profile depicting the hydrological regime (continuous presence of water), and of plants with a  $C_3$  photosynthetic cycle in RJ-02.

Dating of the humin fraction in SOM through <sup>14</sup>C showed different ages between the profiles. In RJ-01, the organic material was dated from 3,351 to 3,699 years (years before the present), while in RJ-02, was from 2,001 to 2,009 years (years before the present). This large difference in age between the two profiles sampled from areas located in proximity to each other highlights the influence of landscape in the maintenance of carbon supplied to form the *Organossolos*. In the RJ-01 profile, the landscape position and slope resulted in poor drainage conditions, while for the RJ-02 profile, they influenced the rate of decomposition through better drainage.

# **CONCLUSIONS**

Based on isotopic analysis, different plant groups ( $C_3$ ,  $C_4$ ) colonized the environment and contributed to the formation of the *Organossolos*.

The influence of algae in the past, along with the presence of  $C_3$  and  $C_4$  plants, is noted on RJ-01, which has SOM dated at 3,280±80 years. The RJ-02 profile has more recent soil organic matter (2,005±5 years), with a higher degree of humification, and a greater quantity of  $C_3$  plants contributing to the soil organic matter.

The upper montane environment and the local topographic conditions determined differentiation of the soils and influenced pedogenesis. They had a direct effect through climate and drainage conditions, and an indirect effect on the decomposition of soil organic matter.

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