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# Paleoenvironmental Characterization of a High-Mountain Environment in the Atlantic Forest in Southeastern Brazil

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**ABSTRACT:** Records of changes in the phytosociological structure of vegetation can be observed more clearly in soils that have more significant accumulation of organic matter, like those occurring in high-mountain environments. The aim of this study was to characterize soils formed in high-mountain environments in the Itatiaia National Park (INP), state of Rio de Janeiro, southeastern Brazil, and to discuss the potential of preserved phytoliths as markers of vegetative history and environmental factors. Four profiles were selected, which were morphologically described and evaluated for their physical and chemical properties. For phytolith analysis and high-resolution determination of the stable carbon isotopes, samples were collected at 0.10 m intervals. The profiles showed highly similar morphological characteristics, with peat deposits and colluvial sediments as source material, produced in the highest parts of the landscape. High-mountain soils in the INP have properties related to high contents of organic matter, like high acidity, low base saturation, and high CEC values due to high H<sup>+</sup> contents. The soils are formed by the addition of plant residues, which accumulate due to the cold and humid climate during most of the year in these environments. The phytolith assemblage had a high frequency of morphotypes characteristic of temperate, cold, and high elevation intertropical regions, especially of Pooideae plants. The phytolith indexes indicated open vegetation environments with a predominance of C<sub>3</sub> grasses, suggesting cold climate conditions, and corroborating the δ<sup>13</sup>C isotopic values. The results of phytolith analysis of the profiles reflected characteristics related to soil genesis. Organism is the main soil formation factor, conditioned by the factors relief (elevation) and climate, which resulted in low temperatures and lead organic matter accumulation.

**Keywords:** soil memory, pedogenesis, Histosols, folist soils, organic matter.

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## INTRODUCTION

The high-mountain environments in the Itatiaia National Park (INP), state of Rio de Janeiro, southeastern Brazil, have been a recurring topic of general considerations and more specific studies on tropical morphogenesis (De Martonne, 1943; Modenesi, 1992; Clapperton, 1993; Modenesi and Toledo, 1993; Modenesi-Gautieri and Nunes, 1998; Santos, 1999; Chiessi, 2004; Lima and Melo, 2013; Marques Neto et al., 2015; Marques Neto, 2016). However, in soil science, there are still few published studies about these environments. This lack is mainly perceived for studies that seek to uncover episodes of the geological-geomorphological past and its variables, aiming to better understand the present, which, in turn, promotes an advance in knowledge regarding the current properties and qualities of soils, thus providing prospects for their best use (Vidal-Torrado et al., 2005).

The climatic oscillations caused by the alternation of glacial and interglacial periods during the Quaternary, a period that covers approximately the last 2.6 million years of Earth's geological history, affected Brazilian territory, including the landscapes of the Mantiqueira Mountain range, where the Massif of Itatiaia is located (Marques Neto et al., 2015). Sedimentary records of climate change can be found in the soils of these environments. These records are characterized by deposits of paleoclimatic value and paleontological and paleobotanical documentation, whose ages are attributed mainly to the late Pleistocene (126,000-11,740 years ago) and Holocene (last 11,740 years). These environments have specific climatic conditions that differ from their surroundings concerning humidity and temperature, where soils with high contents of organic matter (OM) develop due to the lower rates of decomposition associated with low temperatures (Benites et al., 2007; Silva et al., 2009; Weissert and Disney, 2013; Benavides and Vitt, 2014; Cooper et al., 2015; Hribljan et al., 2015; Soares et al., 2016).

Studies aiming at reconstituting past environmental factors can use tools known as proxy indicators (pollen, spores, sponge spicules, phytoliths, among others), which can be interpreted as local records of climate change (Borba-Roschel et al., 2006; Calegari et al., 2013; Coe et al., 2013, 2014; Calegari et al., 2015, 2017a, b; Rasbold et al., 2016; Silva Neto et al., 2018). Sites must be favorable to these types of studies, such as soils with high contents of OM under conditions that can preserve material deposited (Salgado-Labouriau, 1994; Suguio, 1999). One product of such conditions is phytoliths, which are microscopic particles of hydrated silica formed in plant tissues during plant growth (Piperno, 2006) that remain when the plants die and decompose. Through these particles, it is possible to characterize the vegetation; thus, they can be instrumental in paleoenvironmental reconstruction studies.

The INP has areas of Atlantic Forest in several stages, from lush vegetation in different regeneration phases to specific vegetation like high-altitude rocky fields (Benites et al., 2007). These areas have high environmental and ecological importance due to their high carbon storage capacity and the ability to recharge aquifers or even to serve as a substrate for adapted vegetation that is capable of buffering toxic elements (Weissert and Disney, 2013; Cooper et al., 2015). Information on the soil, vegetation, and climate of preserved Atlantic Forest environments, such as the INP, increase the potential for contributing to understanding of all aspects of biodiversity, protection of fauna and flora, climate change, environmental transformations, and water resources. In this context, the aim of this study was to characterize soils formed in high-mountain environments in the INP and to discuss the potential of phytoliths as markers of vegetation history and environmental factors.

## MATERIALS AND METHODS

### Study Area

The study area is in the INP (Figure 1) in the Mantiqueira Mountain range on the border between the states of Rio de Janeiro and Minas Gerais, including the municipalities of

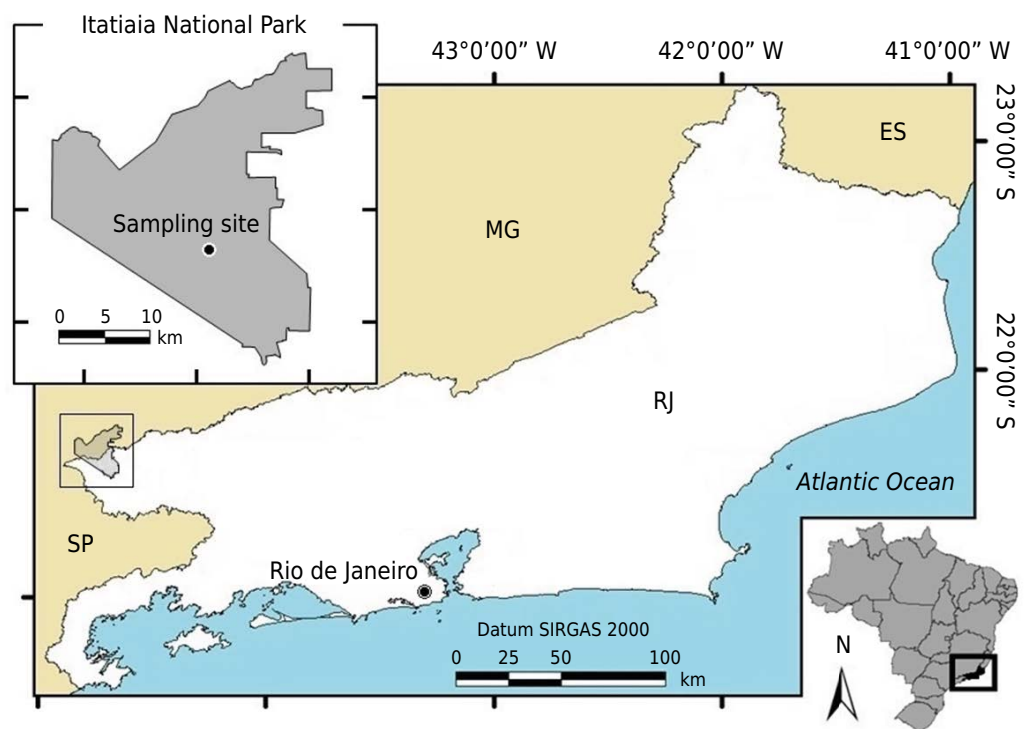
Itatiaia and Resende, in southern Rio de Janeiro, and Bocaina de Minas and Itamonte, in southern Minas Gerais. The INP is a conservation unit managed by the Chico Mendes Institute for Biodiversity Conservation (ICMBio). It is characterized by high, steep terrain, with altitudes ranging from 2,000 to 2,791 m, culminating in the Agulhas Negras Peak (Barreto et al., 2013).

The predominant geological formation in the upper part of the INP is the Itatiaia Alkaline Massif over gneisses of the geological basement that support the Mantiqueira Mountain range. The following types of rock can be found in the Mantiqueira: gneisses, nepheline syenites, quartz syenites, alkaline granites, and magmatic breccia, as well as colluvial and alluvial sediments (Barreto et al., 2013). The vegetation of the upland is described as a high-altitude rock complex, with high endemism of flora and fauna, that is formed by a mosaic of plant typologies associated with the Atlantic Forest. Vegetation in the study area comprises herbaceous plants of graminoid aspect, with predominance of Cyperaceae and Poaceae plants arranged in clumps, as well as plants of other families in smaller numbers (Soares et al., 2016).

The high-altitude part of the INP is characterized by extreme climatic conditions, with possible freezing temperatures, frequent frosts, and high cloudiness (Marques Neto, 2016). In the Köppen climate classification system, it is Cwa, which corresponds to an upland sub-tropical climate, with hot rainy summers and cold dry winters, and average annual temperature of 16 °C and average annual rainfall of 2,300 mm. At the highest point of the INP, the Agulhas Negras Peak, frosts can occur, and the temperature can reach -10 °C in June to August (Barreto et al., 2013).

### Sampling and analyses

The area was chosen from previous field studies based on records of the occurrence of soils with high contents of OM. It was assumed that these soils would offer significant potential for paleoenvironmental studies due to the colder and humid climate of the mountain region. In the selected sites, the soils show no degree of anthropic intervention. According



**Figure 1.** Map of the geographical location of the Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil, and the sampling site.

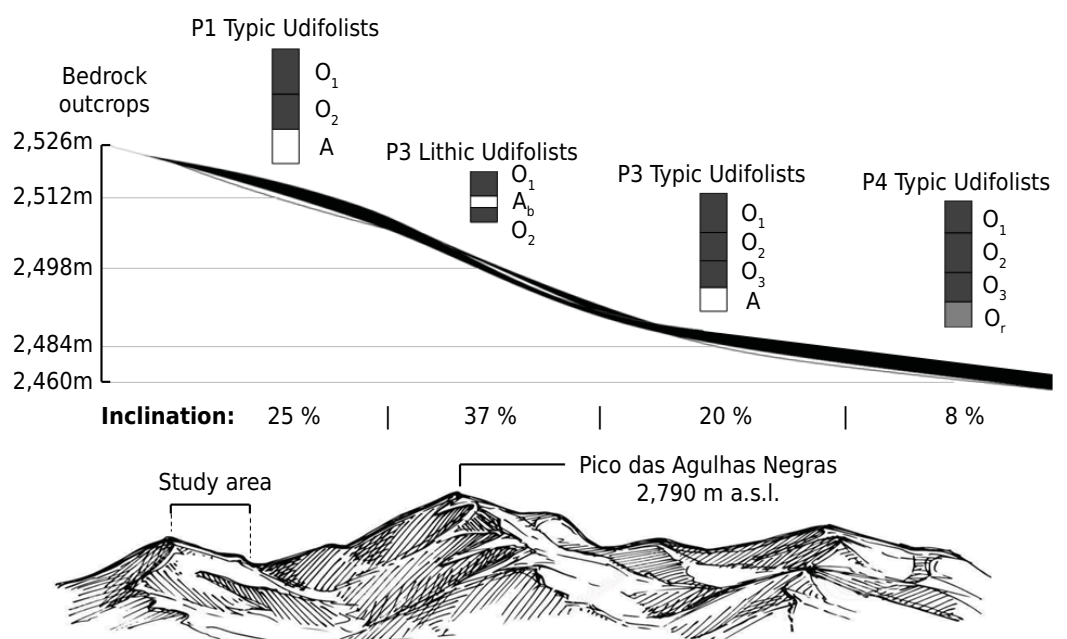
to the INP management plan, areas of preserved and protected vegetation (high altitude fields) are found above 2,000 m. They represent important fragments of Atlantic Forest in southeastern Brazil, with natural wealth still to be discovered (Barreto et al., 2013).

In the study area, a toposequence was selected where four pits were opened in the upper, middle, and lower thirds and in the lowland (Figure 2) for morphological description of the profiles according to the *Manual de Descrição e Coleta de Solo no Campo* - Field Soil Collection and Description Manual - (Santos et al., 2013a). Samples were collected from the respective horizons for soil characterization and classification, and from depths at 0.10 m intervals for phytolith analysis and high-resolution determination of stable carbon isotopes. The samples were first air dried, crushed, and sieved through a 2.0 mm mesh to obtain fine air-dried earth, except for samples that were analyzed by methods requiring different preparation (i.e., phytoliths and isotope ratio).

Analytical determinations followed the methods described in the *Manual de Métodos de Análises de Solos* (Manual of Soil Analysis Methods) (Donagema et al., 2011). Based on the manual, values of pH in water, KCl, and CaCl<sub>2</sub>, exchangeable cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, K<sup>+</sup>), potential acidity (Al+H), and available P extracted with Mehlich-1 were determined. The following indexes were obtained: the sum of bases (S), cation exchange capacity (T), base saturation (V%), and saturation of exchangeable Al (m%). Undisturbed samples were also collected in the profiles through volumetric rings and sent to the laboratory to determine soil bulk density (Bd).

Soil organic carbon and OM contents were determined through the dry combustion method in a muffle furnace (Santos et al., 2013b). The total nitrogen (N) content in the soil samples was quantified by the Kjeldahl method (Tedesco et al., 1995). In the samples of organic composition, tests described in the Brazilian Soil Classification System (SiBCS) (Santos et al., 2013b) were carried out to characterize and classify the histic horizons. The tests included determination of OM and mineral matter (MM) contents, Bd, organic matter density (OMD), minimum residue (MR), the pyrophosphate index (PI), and the Von Post decomposition scale.

For the samples collected at depths, the isotope ratio was quantified by an isotope ratio mass spectrometer (IRMS) (Delta V Advantage) coupled to an IRMS elemental analyzer (Flash EA 2000), both from Thermo Fisher Scientific (Bremen, Germany), at the Carbon



**Figure 2.** Transect with the points of description and collection of profiles in the Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil. (a.s.l. = above sea level).



and Nitrogen Biotransformation Research Laboratory (LABCEN) of the Federal University of Santa Maria, Rio Grande do Sul, Brazil. In these samples, phytoliths were also extracted from the soil according to procedures described in Costa et al. (2010), with modifications, which consisted of low speed spin cycles (1,000 rpm for 5 min) to avoid destruction of more sensitive morphotypes. Morphotypes were identified under an optical microscope using 40 × magnification lenses. On each slide, at least 200 phytoliths with taxonomic and ecological significance were counted and identified according to the International Code for Phytolith Nomenclature (ICPN) (Madella et al., 2005).

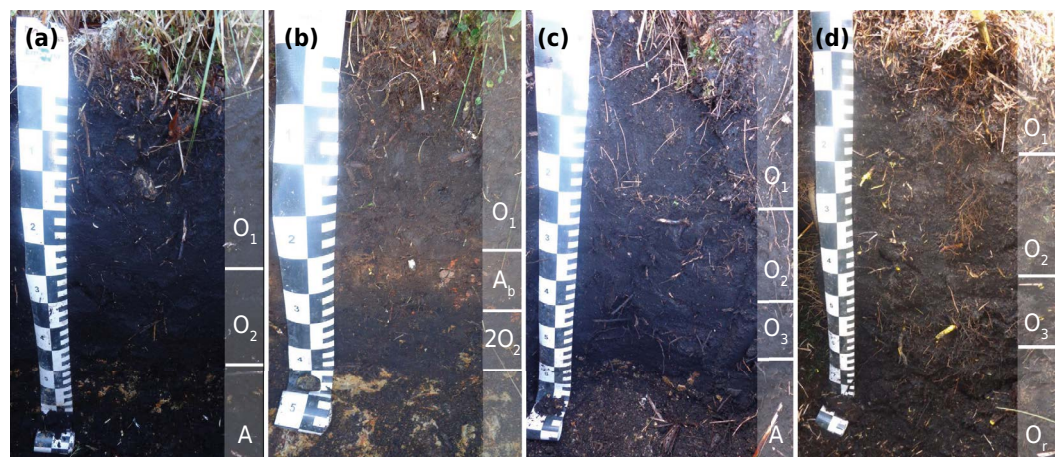
Three phytolith indexes for environmental and taxonomic interpretations were calculated from the assemblies identified. The first was the tree coverage index (D/P) (Alexandre et al., 1997), based on the ratio between Eudicotyledon phytoliths (globular granulate) and the sum of Poaceae (Pooideae, Chloridoideae, Panicoideae, trichomes, and bulliform) phytoliths. The D/P is reliable for tropical regions (Delhon, 2005). The second was the climate index (Ic) (Twiss, 1992), which provides information on the temperature of the study area and is based on the relationship between the number of Pooideae phytoliths divided by the sum of the Pooideae (rondel and trapeziform short), Chloridoideae (saddle), and Panicoideae (bilobate and polylobate) morphotypes × 100. Finally, the bulliform index (Bi%) (Bremond et al., 2005) was used to assess the water stress endured by the grasses and, indirectly, the aridity of the environment in which the phytoliths were formed. In this index, the ratio of bulliform morphotypes (bulliform cuneiform) to the sum of short cells, bulliform, and acicular hair × 100 is determined.

## RESULTS AND DISCUSSION

### Profile characterization

The profiles showed highly similar morphological properties, such as color and moderate to strong structure, differing only in size, with soft consistency (ranging from friable to very friable) (Table 1). A predominantly granular structure was observed, whose development is associated with the capacity of OM in assisting the formation of structural aggregates and their stabilization (Six et al., 2000). Except for P2, all the other profiles were classified as *Organossolo Fólico Sáprico típico* (Typic Udifolists), with predominance of dark colors (N2/) related to high contents of highly decomposed OM (Figure 3).

In the P1 and P3 profiles, the histic horizons are superimposed on mineral horizons with gravel and pebble size fragments, sub-angular and angular, in a semi-consolidated (brittle) stage, with primary minerals observed with the naked eye throughout the soil



**Figure 3.** Profiles collected in the Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil. (a) P1 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists); (b) P2 Profile = *Neossolo Litólico Hístico típico* (Lithic Udifolists); (c) P3 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists); (d) P4 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists).

matrix in the P3 profile. The P2 profile was not as thick as the others and was classified as a *Neossolo Litólico Hístico típico* (Lithic Udifolists), related to its position on a steeper slope. In this profile, a mineral A horizon was observed between the histic horizons, with pedogenetic properties identified as having been developed before the horizon was buried, with common mottling that was small to medium, prominent, and red (2.5YR 4/6).

The source material of the profiles is peat deposits and colluvial sediments, formed at the highest parts of the landscape. At the upper part of the slope where the profiles were collected, there are outcrops of nepheline-syenite alkaline rocks with centimeter sized particles, rich in amphiboles, titanite, and nepheline. A large number of fractures and chemical weathering lead to an infinity of blocks and rolled materials of varied dimensions, deposited along the slope. *Organossolos Fólicos* (Typic Udifolists) are usually associated with a shallow rocky substrate or even adjacent rocky outcrops. When studying the soils associated with rocky outcrops in the Espinhaço and the Mantiqueira Mountain ranges, Benites et al. (2007) found underdeveloped *Organossolos* (Typic Udifolists) together with rocky outcrops and verified that the source materials strongly influenced their properties.

The formation of peat deposits in high-mountain environments, where the climate is humid and cold during most of the year, as in the INP, can occur even in non-hydromorphic conditions through litter accumulation. Several studies show that OM accumulation under well-drained conditions occurs due to reduction in the metabolic activity of microorganisms, disfavoring decomposition of the plant material and leading to accumulation of material and thickening of the organic layer (Zech et al., 1997; Benites et al., 2007; Silva et al., 2013; Bispo et al., 2015; Soares et al., 2016). However, the subprocesses and reactions involved in the formation of the *Organossolos Fólicos* (Typic Udifolists) are still not well understood.

The average organic carbon content in the histic horizons was 160.7 g kg<sup>-1</sup>, with the highest values observed in the P1 profile (Table 2). The degree of decomposition of soil OM evaluated by the von Post and PI analyses, together with rubbed fiber analysis, allowed identification of sapric and hemic materials. In the profiles located in the lower part of the landscape, the

**Table 1.** Morphological properties of the profiles studied in Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil

Hor.	Layer	Munsell color	Structure <sup>(1)</sup>	Soil consistence	Transition <sup>(2)</sup>
m					
P1 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)					
O <sub>1</sub>	0.00-0.27	N2/	gr.	soft, very friable	S. and C.
O <sub>2</sub>	0.27-0.47	N2/	gr.	soft, very friable	R. and C.
A	0.47-0.68 <sup>+</sup>	10YR 2/1	gr.	soft, friable	-
P2 Profile - <i>Neossolo Litólico Hístico típico</i> (Lithic Udifolists)					
O <sub>1</sub>	0.00-0.19	N2/	gr.	soft, very friable	R. and C.
A <sub>b</sub>	0.19-0.28	10YR 3/2	gr.	soft, friable	R. and C.
2O <sub>2</sub>	0.28-0.39	N2/	gr.	soft, very friable	-
P3 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)					
O <sub>1</sub>	0.00-0.23	N2/	gr.	soft, very friable	R. and C.
O <sub>2</sub>	0.23-0.40	N2/	gr. and sbk.	soft, friable	R. and C.
O <sub>3</sub>	0.40-0.56	N2/	gr. and sbk.	soft, friable	R. and C.
A	0.56-0.70 <sup>+</sup>	N2/	gr. and sbk.	soft, friable	-
P4 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)					
O <sub>1</sub>	0.00-0.19	10YR 3/1	gr.	soft, very friable	R. and C.
O <sub>2</sub>	0.19-0.42	N2/	gr.	soft, very friable	S. and C.
O <sub>3</sub>	0.42-0.59	N2/	sbk.	soft, friable	S. and C.
O <sub>r</sub>	0.59-0.74	N2/	gr.	soft, very friable	-

Hor. = horizon. <sup>(1)</sup> gr = granular; sbk = subangular blocky. <sup>(2)</sup> S = smooth; R = rolling; C = clear.

surface horizons exhibited hemic OM, composed of leaves and roots in the intermediate decomposition stage between fibric and sapric OM. The presence of this material suggests that there is a significant addition of plant residues to the soil by the vegetation in this site. In these horizons, the greatest quantities of rubbed fibers were observed - 29 % in the P3 profile and 31 % in the P4 profile. At layers, the OM tends to be more decomposed because it has been in the soil a longer time. This explains the greater degree of decomposition at depths.

Soil bulk density values ranged from 0.23 to 0.82 Mg m<sup>-3</sup> (Table 2). Overall, there was an increase in Bd at depths, and the highest densities occurred in the mineral horizons. In addition to the high contents of OM, the Bd in the *Organossolos* (Typic Udifolists) is also affected by the degree of decomposition of the plant material, with the lowest values observed in the horizons with a lower degree of decomposition. Regarding the content of MM, the values showed uneven distribution within each profile, and no increase or progressive reduction was verified at depths, probably due to the deposition of sediments produced in the upper part of the landscape during OM accumulation.

Concerning chemical characterization, the values of pH in pH(H<sub>2</sub>O) were higher than those in KCl (Table 3), which is probably due to the extraction of organic molecules of carboxyl radicals by the salt solution (KCl). The pH method using KCl is more efficient in characterization of acidity in soils with high contents of OM (Plieski et al., 2004). Contrary to mineral soils, low pH values in organic soils are mainly related to the presence of organic acids. In a study characterizing *Organossolos* (Typic Udifolists) in the INP, Soares et al. (2016) found similar results, with values higher than those quantified in studies performed in similar environments (Scheer et al., 2011; Silva et al., 2013; Könönen et al., 2015). The authors attributed the higher pH values to the alkaline material in the upper part of the INP.

Exchangeable base concentrations (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, K<sup>+</sup>) had higher values in the surface horizons, which was reflected in the SB and V% values. This pattern is likely related to

**Table 2.** Specific analyses for characterization of the profiles studied in Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil

Hor.	Bd	MM	OM	TOC	N	C/N	PI	RF	von Post	
									Index	Material
	Mg m <sup>-3</sup>	%	g kg <sup>-1</sup>					%		
P1 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)										
O <sub>1</sub>	0.46	49.64	287.0	210.9	12.3	16.4	1	24	H9	Sapric
O <sub>2</sub>	0.45	37.46	512.0	182.0	7.8	23.33	1	19	H9	Sapric
A	0.65	84.32	97.3	73.8	2.2	33.53	1		-	-
P2 Profile - <i>Neossolo Litólico Hístico típico</i> (Lithic Udifolists)										
O <sub>1</sub>	0.34	64.95	204.0	149.8	9.1	16.46	1	23	H8	Sapric
A <sub>bf</sub>	0.50	55.73	306.7	56.3	3.2	17.6	2		-	-
2O <sub>2</sub>	0.43	75.57	171.3	146.5	7.6	19.27	0	16	H8	Sapric
P3 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)										
O <sub>1</sub>	0.26	57.25	234.0	183.4	8.2	22.36	1	25	H7	Hemic
O <sub>2</sub>	0.38	64.36	199.3	151.4	6.7	22.58	1	17	H8	Sapric
O <sub>3</sub>	0.41	67.68	184.7	141.4	7.1	19.91	0	15	H8	Sapric
A	0.82	86.40	84.7	70.0	1.5	46.65	3		-	-
P4 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)										
O <sub>1</sub>	0.23	52.78	277.3	195.2	10.7	18.24	1	25	H7	Hemic
O <sub>2</sub>	0.40	73.23	161.3	125.8	4.9	25.67	3	17	H8	Sapric
O <sub>3</sub>	0.37	55.90	198.0	140.8	8.3	16.96	3	23	H8	Sapric
O <sub>r</sub>	0.38	67.59	187.3	140.3	7.6	18.42	3	11	H8	Sapric

Bd = bulk density; MM = mineral material; OM = organic matter; TOC = total organic carbon; N = nitrogen; C/N = carbon to nitrogen ratio; PI = sodium pyrophosphate index; RF = percentage of rubbed fibers. Soil samples were analyzed according to the methods proposed by Santos et al. (2013 b).



the continuous contribution of OM by the local vegetation described as high-altitude rock complex (Benites et al., 2007), composed of herbaceous plants and grasses arranged in dense clumps. Values of  $Mg^{2+}$  higher than  $Ca^{2+}$  may be related to the chemical composition of the parent material. Similar results were found by Soares et al. (2016) for *Organossolos* (Typic Udifolists) in the upper part of the INP. The contents of available P were highly variable, ranging from 0.6 to 27  $mg\ kg^{-1}$ . In a study evaluating soils similar to these, Valladares et al. (2008) considered that values of available P below 10  $mg\ kg^{-1}$  are low for soils with high contents of OM. Therefore, in general, the profiles studied can be characterized as having low P contents since 86 % of the horizons are below this limit.

The high values of CEC (T value) are due to the high  $H^+$  contents, which predominated in the sorptive complex due to the high contents of OM (Pereira et al., 2005). The Al contents ranged from 0.73  $cmol_c\ kg^{-1}$  in the mineral  $A_b$  horizon to 8.93  $cmol_c\ kg^{-1}$  in the  $O_1$  histic horizon of the P1 profile. In the *Organossolos* (Typic Udifolists), most of the exchangeable acidity is due to the  $H^+$  contents coming from organic acids and hydrolysis of other compounds, unlike what occurs in mineral soils, where this acidity is due to the hydrolysis of  $Al^{3+}$  ions (Ebeling et al., 2008; Silva et al., 2008).

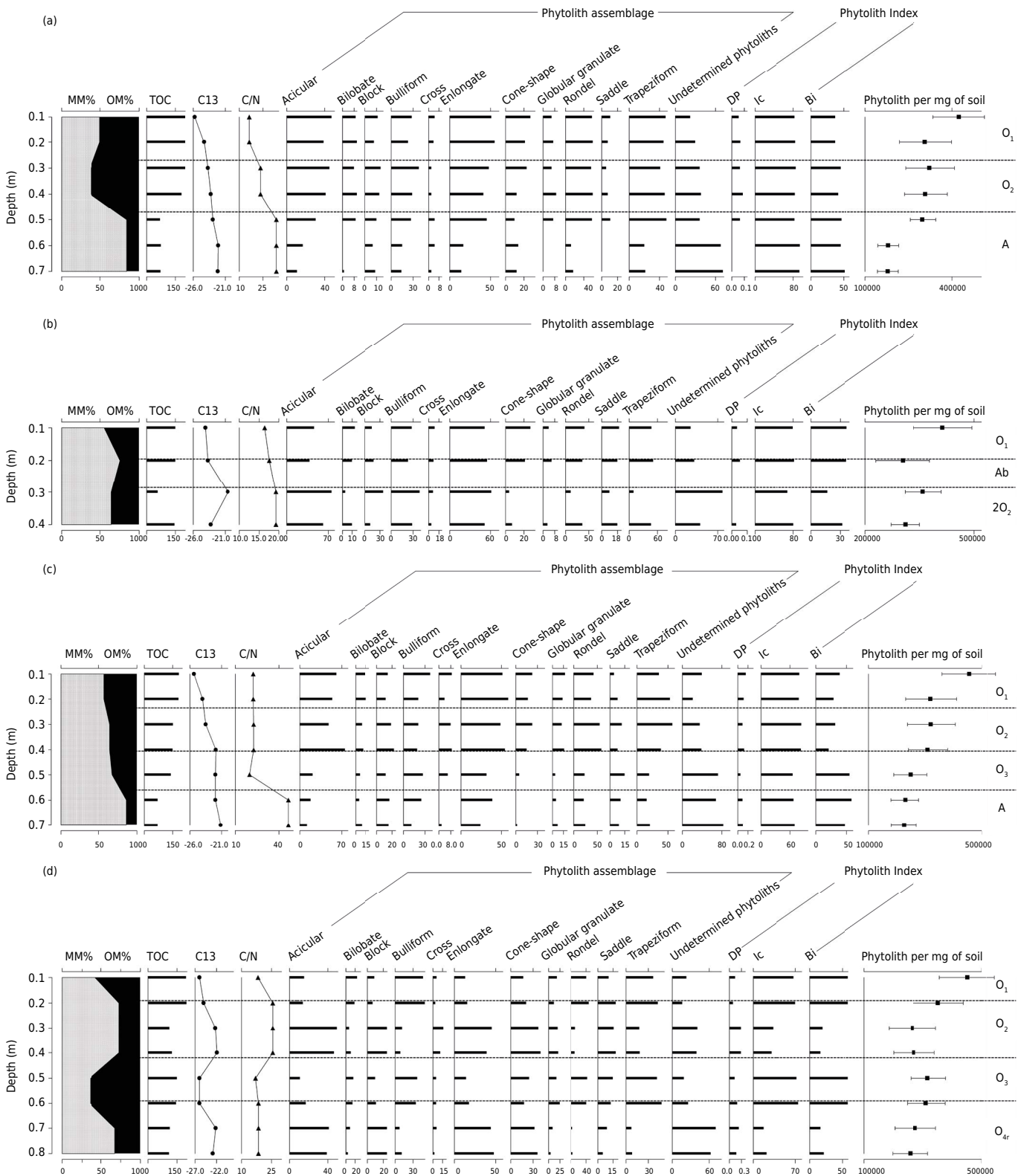
### Phytolith analysis in the soil

The distribution of the main morphotypes of the phytoliths identified and results of the isotope ( $\delta^{13}C$ ) ratio are shown in figure 4. Phytolith analysis revealed assemblages with relevant morphotype varieties and quantities within and between profiles. The most abundant morphotypes belonged to the Poaceae family (elongate, acicular hair cell, rondel, and trapeziform). Phytoliths of the saddle morphotype, produced by Chloridoideae, and cross morphotype, produced by Panicoideae, were observed among those that are formed in short cells. Among phytoliths with taxonomic significance, cone-shaped morphotypes, characteristic of the Cyperaceae family, were also observed. These results

**Table 3.** Chemical properties of the profiles studied at Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil

Hor.	pH (1:2.5)			$Ca^{2+}$	$Mg^{2+}$	$K^+$	$Na^+$	SB	$Al^{3+}$	$H^+$	T	V%	m%	P
	$H_2O$	KCl	$CaCl_2$											
cmol <sub>c</sub> kg <sup>-1</sup>														
mg kg <sup>-1</sup>														
P1 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)														
$O_1$	4.62	3.85	3.77	0.48	1.34	0.02	0.01	1.9	8.93	58.23	69.01	3	13	8
$O_2$	4.43	4.06	3.98	0.28	1.12	0.01	0.01	1.4	6.59	51.09	59.10	2	11	1
A	4.78	4.32	4.18	0.27	1.41	0.01	0.01	1.7	3.32	28.51	33.52	5	10	8
P2 Profile - <i>Neossolo Litólico Hístico típico</i> (Lithic Udifolists)														
$O_1$	4.87	4.16	4.19	0.34	1.18	0.03	0.01	1.6	2.89	13.45	17.9	9	16	15
$A_{bf}$	4.64	4.58	4.42	0.12	1.56	0.01	0.01	1.7	0.73	26.83	29.25	6	2	27
$2O_2$	4.53	4.28	4.15	0.45	0.92	0.01	0.01	1.4	3.64	36.37	37.76	4	10	10
P3 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)														
$O_1$	4.46	4.08	3.96	0.36	1.09	0.02	0.01	1.5	5.9	46.74	48.22	3	12	7
$O_2$	4.00	4.22	4.06	0.24	1.13	0.01	0.01	1.4	4.99	46.37	47.76	3	10	7
$O_3$	4.77	4.25	4.18	0.18	0.92	0.01	0.01	1.1	3.67	41.27	42.38	3	9	6
A	4.46	4.44	4.27	0.07	1.31	0.01	0.01	1.4	2.07	23.12	24.51	6	8	8
P4 Profile - <i>Organossolo Fólico Sáprico típico</i> (Typic Udifolists)														
$O_1$	5.15	4.33	4.23	1.05	1.51	0.10	0.01	2.7	2.71	23.66	29.05	9	9	9
$O_2$	4.88	4.36	4.28	0.52	1.47	0.02	0.01	2.0	2.56	25.44	30.03	7	9	9
$O_3$	4.99	4.34	4.24	0.74	1.20	0.03	0.01	2.0	2.29	25.36	29.64	7	8	9
$O_r$	4.80	4.43	4.30	0.38	1.41	0.02	0.01	1.8	1.62	25.37	28.81	6	6	9

Hor. = horizon; SB = sum of bases; T = cation exchange capacity; V = base saturation; m = aluminum saturation. Soil samples were analyzed according to the methods proposed by Donagema et al. (2011).



**Figure 4.** Frequency values of phytoliths identified and indices analyzed in the profiles collected in Itatiaia National Park, state of Rio de Janeiro, southeastern Brazil. (a) P1 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists); (b) P2 Profile = *Neossolo Litólico Hístico típico* (Lithic Udifolists); (c) P3 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists); (d) P4 Profile = *Organossolo Fólico Sáprico típico* (Typic Udifolists).

corroborate the study by Soares et al. (2016) that characterized *Organossolos* (Typic Udifolists) in the upper part of the INP. According to the authors, these soils represent dense accumulations of OM with a high degree of humification, originating from the same type of herbaceous vegetation with a graminoid aspect, with predominance of Cyperaceae and Poaceae plants.

### **P1 Profile - *Organossolo Fólico Sáprico típico* (Typic Udifolists)**

The frequency of phytoliths in the P1 profile had a pattern of decreasing accumulation at depths, a result commonly observed in studies of soil phytoliths (Alexandre et al., 1997; Lepsch and Andrade Paula, 2006; Calegari et al., 2013). In the O<sub>1</sub> and O<sub>2</sub> horizons, the phytolith assemblage is predominantly composed of phytoliths produced by plants of the Poaceae family, acicular hair cell (~18 %), elongate (~17 %), and bulliform (~13 %), with the number of rondel (~16 %) and trapeziform (~15 %) morphotypes standing out, characteristic of temperate, cold, and high elevation intertropical regions. The frequency of woody dicotyledon phytoliths ranged from 2 to 3 % in the histic horizons, but these phytoliths were absent at the base of the profile. In the A horizon, at depths of 0.6 m and 0.7 m, in addition to the lower frequency of phytoliths, a higher number of phytoliths was altered/subjected to taphonomic processes (~35 %).

The values of the D/P index were low, indicating an environment with open vegetation (Alexandre et al., 1997, 1999; Barboni et al., 1999, 2007). At depths of 0.6 and 0.7 m, this index could not be calculated, due to the absence of dicotyledon phytoliths. The Ic ranged from 82.7 to 85.7 %, with the highest values observed in the A horizon. The high Ic values indicate the predominance of C<sub>3</sub> grasses, suggesting cold climate conditions (Twiss, 1987, 1992). Concerning the Bi index values, a tendency to increase at depths was observed, with average values of 37.3 % in the O<sub>1</sub> horizon and 49.7 % in the A horizon. High Bi index values may indicate local water stress or a high transpiration rate, leading to high production of bulliform phytoliths by plant tissues (Lorente et al., 2015).

The results of phytolith analysis of the P1 profile reflect characteristics related to soil genesis, conditioned by topographic (altitude) and climate factors, which result in low temperatures, leading to OM accumulation. The lower frequency of phytoliths at the base of the profile, as well as the higher frequency of unidentified phytoliths, may be related to the processes of loss and transport of material by erosion. The modeling of the Itatiaia highland fields is essentially erosive and characterized by intense dissection. Evidence of this process is the gravel and pebble size fragments, sub-angular and angular, in the semi-consolidated stage observed in the A horizon. The chronology of these deposits is attributed to the late Pleistocene and early Holocene, found at a lower level of ramps extending over the peat deposits, with a gravelly appearance and sharp color contrast between the dark matrix and the whitish granules and pebbles. These characteristics suggest the performance of different processes in the evolution of the slopes, likely related to climatic variations during the Quaternary (Modenesi, 1992; Modenesi and Toledo, 1993, 1996; Marques Neto et al., 2015, 2016).

### **P2 Profile - *Neossolo Litólico Hístico típico* (Lithic Udifolists)**

In the P2 profile, phytolith analysis indicated the presence of a buried A horizon, with an increase in the number of phytoliths at the depths evaluated that comprise the A<sub>b</sub> horizon (Figure 3b). The phytolith assemblage also comprises phytoliths produced by plants of the Poaceae family, acicular hair cell (~16 %), elongate (~16 %), and bulliform (~10 %). The rondel (~16 %) and trapeziform (~15 %) morphotypes typical of the Pooideae subfamily and characteristic of cold environments, like high intertropical elevations, together represented 30 % of the phytoliths identified in the A<sub>b</sub> horizon. The number of woody dicotyledon phytoliths was low, with 2 % as the maximum value in the O<sub>1</sub> horizon; they were absent at the 0.3 m depth. In the transition between the A<sub>b</sub> and 2O<sub>2</sub> horizons (~0.3 m depth), besides the higher frequency of phytoliths, the highest frequency (19 %) of phytoliths that were altered/subjected to taphonomic processes was observed among the depths evaluated throughout the profile.

Similar to the P1 profile, the D/P index values were low, indicating an environment with open vegetation. The Ic ranged from 69.5 to 82.4 %, with the lowest values observed in the A<sub>b</sub> horizon. The Bi also had a lower value (~17 %) in the A<sub>b</sub> horizon, indicating a period of lower

local water stress. The results of phytolith analysis, associated with the  $\delta^{13}\text{C}$  isotopic values, indicated a more humid climate transition during soil formation. Soil genesis is characterized by the addition of colluvium, probably caused by displacement of material in wetter and perhaps warmer climates, capable of weathering the slope materials and providing the water necessary to trigger the erosive process, likely in the late Pleistocene (126,000-11,740 years ago), following the glacial maximum (Modenesi, 1992). At the beginning of the Holocene (in the last 11,740 years), land mass movements may have caused transport of the pedological cover for a short distance in areas of unstable topography and some altitude, as in the coastal and Mantiqueira Mountain ranges (Silva and Vidal-Torrado, 1999).

The phytolith assemblage had a high percentage of morphotypes characteristic of temperate, cold, and high elevation intertropical regions, especially of Pooideae  $\text{C}_3$  plants (Barboni et al., 1999). The  $\delta^{13}\text{C}$  isotopic values corroborate this result, indicating a predominance of  $\text{C}_3$  plants. Above a certain altitude, a clear limit of change occurs in forest vegetation, which transforms to more open vegetation, and then to vegetation without trees where only forage and grass species can develop (Madella et al., 2011). Between 20,000 and 10,000 years ago, climatic conditions in southern and southeastern Brazil were colder and the peaks became open and exposed regions that were colonized by field vegetation that was fire-tolerant and adapted to frequent droughts and frosts (Oliveira et al., 2005).

### **P3 Profile - *Organossolo Fólico Sáprico típico* (Typic Udifolists)**

The phytolith assemblage of the P3 profile is composed of the same morphotypes identified in the P1 and P2 profiles, with a high percentage of phytoliths produced by plants of the Poaceae family. In the histic horizons, the assemblage was predominantly composed of the morphotypes acicular hair cell (~17%), elongate (~15%), and bulliform (~10%). The rondel (~13%) and trapeziform (~12%) morphotypes, which characterize cold and temperate climate environments, were also observed. The frequency of woody dicotyledon phytoliths was higher than that observed in the previous profiles, ranging from 2% (0.6 m depth) to 5% (0.1 m depth). Similar to the P1 profile, a higher frequency (~32%) of phytoliths that were altered/subjected to taphonomic processes was observed in the A horizon at depths of 0.6 and 0.7 m, in addition to the lower frequency of phytoliths. The higher frequency of phytoliths in the histic horizons, as well as the lower proportion of indeterminate phytoliths, is related to conditions favorable to preservation in the deposition environment.

The phytolith indexes indicate an environment with open vegetation and cold climate conditions with little variation in moisture. The D/P index had low values throughout the profile, which reflects an environment with more open vegetation. Similar results were found by Parolin et al. (2017) in a paleoenvironmental study on the middle Holocene using phytoliths found in the Cadeado Mountain range, state of Paraná, southern Brazil. According to Lorente et al. (2015), this index still lacks calibration in Brazilian landscapes since the values obtained for areas of Dense Ombrophilous Forest, Mixed Ombrophilous Forest and Cerrado have been smaller when compared to the African continent. The Ic ranged from 78.7 to 82.8%, with the highest values observed in the depths close to the surface, reflecting the presence of Pooideae plants. This subfamily is characteristic of temperate, cold, and high elevation intertropical regions, producing mainly phytoliths of the *rondel* and *trapeziform* morphotypes.

According to Oliveira et al. (2005), peat bogs of high-mountain environments are approximately 9,000 years old. When characterizing *Organossolos* (Typic Udifolists) in a high-mountain environment of the INP, Soares et al. (2016) found ages between 3,699 and 2,009 BP for dating with  $^{14}\text{C}$  of OM from the soil. Around 9,700 to 8,200 BP, the climate in the summits/extensions of the Mantiqueira Mountain range (Itatiaia) would have been wetter and colder than the current one, and between 8,200 and 5,400 years, the climate would have become warmer and drier (Suguio, 1999). The peat bogs are unique environments for studies aiming to understand landscape evolution according

to climatic changes since they hold records of the temporal and spatial dynamics of the vegetation, being formed of *Organossolos* (Typic Udifolists) with high indicator potential due to the high contents of carbon and an anoxic environment. These conditions preserve the OM and organisms that have been deposited in the past (Horák et al., 2011).

#### **P4 Profile - *Organossolo Fólico Sáprico típico* (Typic Udifolists)**

The main morphotypes identified in the P4 profile were also grasses, especially those formed in larger cells, like the elongate (~13 %), acicular (~12 %), and bulliform (~10 %). Among the morphotypes that are formed in short cells, a higher frequency of saddle phytoliths produced by Chloridoideae plants, and cross by Panicoideae was observed. Among the phytoliths with taxonomic meaning, the *rondel* (~10 %) and *trapeziform cell* (9 %) morphotypes were also found. The highest frequencies of woody dicotyledon phytoliths were observed among the profiles in P4, with an average of 7 % in the O<sub>2</sub> horizon and 9 % in the O<sub>r</sub> horizon. The frequency of phytoliths at depths exhibited an alluvial pattern, with higher and lower concentration phases, indicating an alternation of depositional cycles, probably related to the position of the profile at the lower part of the slope. The number of phytoliths that were altered/subjected to taphonomic processes showed a similar pattern, with higher values for the O<sub>2</sub> and O<sub>r</sub> horizons.

Interpretation of the phytolith indexes indicated the most significant changes in the vegetation formation dominant in these areas in comparison to the current vegetation, based on the mixture of characteristic patterns. In the O<sub>2</sub> and O<sub>r</sub> horizons, the phytolith assemblage is predominantly composed of Poaceae (elongate and acicular) phytoliths and the cone shape morphotype, indicating the presence of Cyperaceae plants (Piperno, 2006; Rasbold et al., 2011), which are characteristic of moist environments. The highest frequency values were also observed for the cross and bilobate phytoliths, which are characteristic of the Panicoideae subfamily of hot and humid environments. The Ic and Bi indexes indicate wetter climate phases, and the  $\delta^{13}\text{C}$  isotopic values reveal the presence of C<sub>4</sub> plants. The highest values of C/N ratio and the lowest concentrations of OM were also detected. The gravel-sized, sub-angular, and sub-angular fragments with different degrees of alteration observed in these horizons suggest reworking by surface processes, with the inclusion of rock fragments during downslope transport.

In contrast, the O<sub>1</sub> and O<sub>3</sub> horizons had the highest concentrations of phytoliths of Pooideae plants, characteristic of temperate, cold, and high elevation intertropical regions, mainly with *rondel* (~19 %) and *trapeziform* (~17 %) phytoliths. The D/P index values were lower, indicating an environment with more open vegetation (Alexandre et al., 1999; Barboni et al., 1999). The high Ic values (~73.2 %) reveal the predominance of C<sub>3</sub> grasses, suggesting cold climate conditions (Twiss 1987, 1992). The  $\delta^{13}\text{C}$  isotopic values corroborate this result, indicating a predominance of C<sub>3</sub> plants. The high values of the Bi index reflect local water stress or high transpiration rate, indicating a drier climate phase with high production of bulliform phytoliths. In these horizons, the highest concentrations of OM were also detected, indicating a phase with characteristics favorable to OM accumulation.

The oscillations caused by the alternation of glacial and interglacial periods during the Quaternary affected Brazilian territory, including the INP highlands. These effects, superimposed on ongoing neotectonics processes, have considerably increased the complexity of morphogenesis. The sedimentary record of changing climates exists mainly in the form of colluvial ramps and alluvial packets, in addition to marsh sediments in high altitude plains, whose ages are attributed mainly to the late Pleistocene and Holocene (Marques Neto, 2012; Marques Neto et al., 2015). From more stable and perhaps warmer wetter climates favoring regolith change, there may have been a shift to intensification or concentration of rainfall (Modenesi and Toledo, 1993), which may be related to phytolith records observed in the profile.



This study represents the first steps of paleoenvironmental characterization of the regions the analyzed profiles are in. However, more comprehensive future research, including correlation with other proxies and efforts at dating may result in more detailed and precise information concerning the paleoenvironmental changes that occurred in the study areas.

## CONCLUSIONS

High-mountain soils in the Itatiaia National Park have properties related to high concentrations of organic matter, such as high acidity, low base saturation, and high CEC values, due to high H<sup>+</sup> contents. The soils are formed by the addition of plant residues, which accumulate due to the cold and wet climate during most of the year in these environments.

The phytolith assemblage had a high frequency of morphotypes characteristic of temperate, cold, and high elevation intertropical regions, especially of Poideae plants. The phytolith indexes indicated open vegetation environments with a predominance of C<sub>3</sub> grasses, suggesting cold climate conditions and corroborating the δ<sup>13</sup>C isotopic values.

The results of phytolith analysis of the profiles reflected characteristics related to soil genesis as the main soil formation factor, conditioned by topographic (altitude) and climate factors, which resulted in low temperatures, leading to organic matter accumulation.

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