

Mineralogical, chemical and electrochemical attributes of soils

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Edited by: Paulo Cesar Sentelhas

Received March 03, 2020

Accepted June 12, 2020

ABSTRACT: A set of attributes endows the soils with distinctive characteristics and astute understanding is required in order to formulate suitable strategies for soil management. The aim of this study was to physically, chemically and mineralogically characterize samples of the main soil classes in Minas Gerais, Brazil, determine the point of zero salt effect (PZSE) and the point of zero charge (PZC), and ascertain the correlation between these factors and soil attributes. The soils evaluated presented different textural classes ranging from loamy sand (Entisol) to very clayey (some Oxisols and Ultisols). The soils differed substantially in terms of fertility, presenting a range from dystrophic (low fertility, base saturation < 50 %) to eutrophic character (fertility, base saturation ≥ 50 %), even within the same soil class, such as the Oxisols, which suggests the concurrence of the parent material. Highly weathered soils are predominant in Minas Gerais and these soils are composed predominantly of kaolinite, gibbsite, goethite and hematite. Traces of hydroxy-Al interlayered vermiculite and illite were also found in the Oxisols, Ultisols and Inceptisols. A correlation between the PZSE and the PZC in the A horizon was observed. A high degree of correlation was observed between the PZC and the exchangeable aluminum and the ratio of iron obtained by ammonium oxalate and dithionite-citrate (Fe_o/Fe_d) in both the A and B horizons of soil classes. The results obtained reinforce the importance of knowledge of soil attributes to the adoption of practices such as the management of phosphate fertilization in clayey soils and liming in soils rich in aluminum.

Keywords: soil attributes, weathered soils, secondary minerals, mineralogical composition, point of zero charge

Introduction

Due to the soil diversity in Brazil, soil characterization is essential to better agricultural or environmental usage and subsequent management. Mineralogical studies are very important to understanding the physical and chemical properties of the soil (Camargo et al., 2008; Almeida et al., 2021). Phosphorous adsorption is mainly attributed to gibbsite, goethite and hematite, the predominant minerals in highly weathered soils (Fontes and Weed, 1996; Schaefer et al., 2008; Fontes, 2012). Electrochemical properties of soils are fundamental to an understanding of the physico-chemical phenomena (Camêlo et al., 2018) that affect soil fertility, availability of nutrients for plants, phytoavailability and metal mobility (Fontes and Alleoni, 2006).

The soils of Minas Gerais represent a large part of those most commonly found in Brazil, such as Oxisols, Ultisols, and Inceptisols. The climatic conditions existing in the state favor the formation and stability of kaolinite, in which the Oxisols and Ultisols stand out.

The factors that favor the formation of kaolinite are those related to warm and humid climate conditions, with moderate drainage, without excessive leaching and low pH (Fontes et al., 2001). These authors mention that under moderate drainage and leaching conditions, the tendency is towards a strong loss of cations and moderate loss of silica, which allows for the formation of kaolinite. Furthermore, in drainage conditions that favor

very strong leaching an almost complete desilication can occur, favoring the formation of aluminum (Al) and iron (Fe) oxy-hydroxides. Thus, kaolinite, Fe and Al oxy-hydroxides are the most common constituents found in tropical soils. They exert great influence on certain physical and chemical characteristics of soils, such as aggregate stability and ion exchange reactions, in addition to being determinant in their pigmentation.

Thus, the study of soil attributes provides significant information on the weathering process, losses of nutrients and organic matter, availability of nutrients for plants, and potential environmental changes among others. Consequently, this study aimed to physically, chemically, electrochemically and mineralogically characterize samples of the main soil classes in the state of Minas Gerais, Brazil.

Materials and Methods

Soil samples

Twenty-three representative profiles of the main soil classes in the state of Minas Gerais, Brazil (Figure 1), were selected at depths of 0 to 20 cm and 50 to 70 cm (Embrapa, 2013). After collection, the soil samples were air-dried, gently crumbled and sieved through a 2-mm mesh to obtain the air-dried fine earth (ADFE).

The soils were classified according to the Brazilian System of Soil Classification (Embrapa, 2013) and up to the category level of the subgroup (Table 1) based on Soil Taxonomy (Soil Survey Staff, 2014).

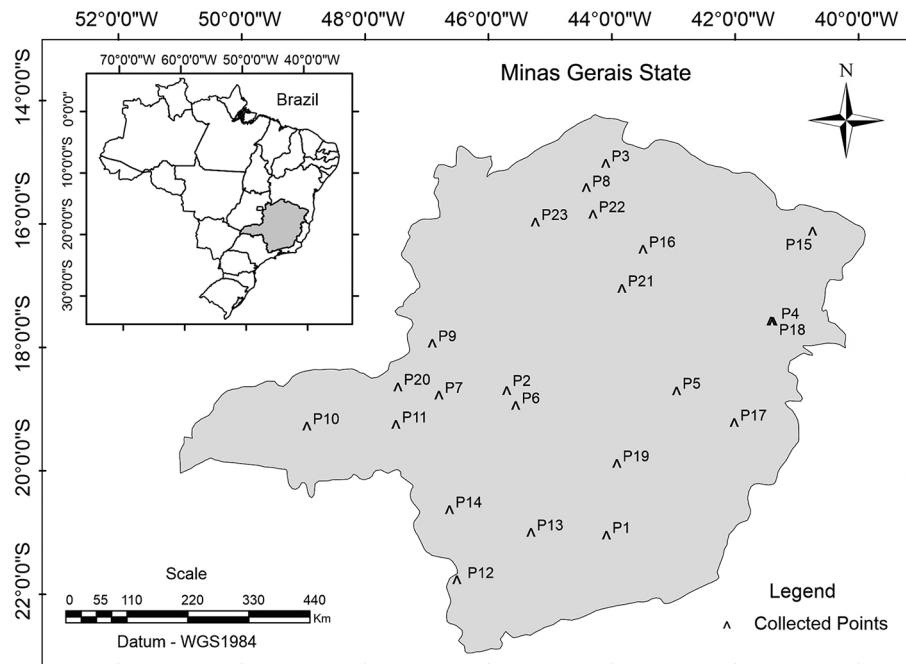


Figure 1 – Location of the collected soils in the state of Minas Gerais.

Table 1 – Soil classification according to the Brazilian System of Soil Classification (SiBCS) and U.S. Soil Taxonomy (Soil Survey Staff, 2014).

Soil Id.	Brazilian System of Soil Classification	Soil Taxonomy	Location	Elevation (m)	Geographic coordinates	
					Latitude	Longitude
P1	Latossolo Vermelho-Amarelo distrófico	Acrudox	Prados	979	-44°05'06.79"	-21°06'31.32"
P2	Latossolo Vermelho-Amarelo distrófico	Acrudox	Capão	1032	-45°42'01.39"	-18°46'05.26"
P3	Latossolo Vermelho-Amarelo eutrófico	Eutruxox	Itacarambi	445	-44°05'47.95"	-15°05'09.75"
P4	Latossolo Vermelho-Amarelo distrófico	Hapludox	Near to Topázio	306	-41°24'53.64"	-17°38'25.04"
P5	Latossolo Vermelho-Amarelo distrófico	Hapludox	Guanhães	778	-42°56'48.60"	-18°46'22.19"
P6	Latossolo Vermelho distrófico	Acrudox	BR-354	886	-45°33'14.00"	-19°00'25.70"
P7	Latossolo Vermelho distrófico	Hapludox	Guimarânia	915	-46°47'56.67"	-18°50'21.82"
P8	Latossolo Vermelho eutrófico	Eutruxox	Januária	434	-44°24'37.12"	-15°28'35.71"
P9	Latossolo Vermelho epieutrófico	Hapludox	Vazante	654	-46°54'26.77"	-18°00'02.09"
P10	Latossolo Vermelho epieutrófico	Hapludox	Prata	631	-48°56'21.79"	-19°20'35.69"
P11	Latossolo Vermelho distrófico	Hapludox	Santa Juliana	910	-47°29'50.89"	-19°18'59.71"
P12	Latossolo Bruno distrófico	Hapludox	Poços de Caldas	1530	-46°34'04.46"	-21°48'21.87"
P13	Argissolo Vermelho-Amarelo epieutrófico	Paleudults	Porto dos Mendes	864	-45°18'26.32"	-21°03'57.78"
P14	Argissolo Vermelho-Amarelo eutrófico	Paleudults	Passos	745	-46°37'44.03"	-20°41'43.90"
P15	Argissolo Vermelho-Amarelo eutrófico	Paleustults	Almenara	205	-40°44'35.59"	-16°11'07.84"
P16	Argissolo Vermelho-Amarelo epieutrófico	Paleustults	Francisco Sá-Montes Claros	650	-43°29'27.97"	-16°28'43.32"
P17	Argissolo Vermelho-Amarelo eutrófico	Hapludults	Tarumirim	318	-42°00'38.96"	-19°17'06.47"
P18	Argissolo Vermelho-Amarelo distrófico	Paleustults	Topázio	347	-41°23'01.28"	-17°38'37.14"
P19	Cambissolo Háplico Tb distrófico	Dystrudepts	Metropolitan region of BH	751	-43°54'56.72"	-19°56'48.98"
P20	Cambissolo Háplico aluminico	Dystrudepts	Monte Carmelo	890	-47°27'55.86"	-18°42'30.34"
P21	Cambissolo Háplico Tb distrófico	Dystrudepts	Montes Claros-Bocaiúva	676	-43°49'54.86"	-17°06'40.38"
P22	Cambissolo Háplico Tb eutrófico	Haplustepts	Lontra	602	-44°18'16.08"	-15°54'33.43"
P23	Neossolo Quartzarênico ortico	Quartzipsamments	Pintópolis	499	-45°14'07.14"	-16°02'15.98"

Physical analysis

Texture, water-dispersible clay (WDC), and the degree of flocculation (DF) were determined (Embrapa, 2017). For WDC analysis, 10 g of ADFE were weighed

in a container with 200 mL of deionized water. These recipients were capped and agitated for 16 h at 50 rpm. The sedimentation time was calculated based on Stokes' Law.

Soil fertility assessment

The following analyses were performed: pH in water and 1 mol L⁻¹ KCl; total organic carbon (TOC) (Yeomans and Bremner, 1988); available phosphorous (P), sodium (Na⁺), and potassium (K⁺) after extraction with 0.5 mol L⁻¹ HCl + 0.0125 mol L⁻¹ H₂SO₄ (Mehlich-1); calcium (Ca²⁺) and magnesium (Mg²⁺) by atomic absorption spectroscopy and Al³⁺ by titration after extraction with 1 mol L⁻¹ KCl; potential acidity (H + Al) by titration after extraction with 0.5 mol L⁻¹ Ca(CH₃COO)₂ at pH 7.0, and remaining P (Alvarez and Fonseca, 1990). The micronutrients iron (Fe) and manganese (Mn) were extracted by means of a chelating solution (DTPA), and the chemical elements were determined by atomic absorption spectrophotometry. Boron (B) was extracted with hot water and determined by colorimetry. All chemical analyses were based on the Brazilian Agricultural Research Corporation's methods (Embrapa, 2017).

Mineralogical analyses

Firstly, the clay and silt fractions were dispersed and separated by sedimentation and the sand fraction segregated by sieving from ADFE (Embrapa, 2017).

X-ray diffraction (XRD) analyses were conducted on sand, silt and clay fractions on an X'pert PRO diffractometer (CoK α radiation) in the ranges of 4 to 50 °2 θ (natural slides) and 5 to 30 °2 θ (clay slides with treatments), with intervals of 0.02 °2 θ to 1 step s⁻¹, voltage of 40 kV, and current of 30 mA. The deferrified slides were prepared both with and without treatment. The samples were saturated with 1.0 mol L⁻¹ KCl and MgCl₂ solutions. KCl-impregnated slides were read before and after heating in a muffle at 550 °C for 3 h. In MgCl₂-impregnated slides, the reading was taken before and after adding one drop of glycerol.

The Fe forms were determined by the methods of dithionite-citrate (Fe_d) (Coffin, 1963), and 0.2 mol L⁻¹ ammonium acid oxalate at pH 3.0 (Fe_o) (McKeague and Day, 1966). The Fe was quantified by an atomic absorption spectrophotometer.

Thermogravimetric determinations

The thermogravimetric analyses (TGA) were conducted by a Shimadzu TGA 50 Thermal Analyzer. For this process, the samples were placed in an alumina cell. The thermobalance operated with a constant flow of nitrogen atmosphere and the analysis temperature range was between the ambient temperature and 800 °C, at a heating rate of 10 °C min⁻¹.

Point of zero Charge

The point of zero charge (PZC) was estimated from the pH values, according to the following equation: PZC = 2 pH KCl - pH H₂O (Keng and Uehara, 1974).

Point of Zero Salt Effect

The point of zero salt effect (PZSE) was determined by potentiometric titration (Fontes et al., 2001) by the

traditional method with modifications (Raij and Peech, 1972). A mass of 4 g of each soil sample was placed in 50 mL containers with 12.50 mL sodium nitrate solution at concentrations of 0.002, 0.02, and 0.2 mol L⁻¹. The samples were acidified using increasing volumes of acid solution (0.5, 1.0, 2.0, and 3.0 mL 0.1 mol L⁻¹ HCl) and titrated with a basic solution (0.5, 1.0, 2.0, and 3.0 mL 0.1 mol L⁻¹ NaOH). Subsequently, distilled water was added to obtain a final volume of 25 mL in each container and dilution of the sodium nitrate concentration to 0.001, 0.01, and 0.1 mol L⁻¹. The material was sealed, stirred, and allowed to stand for 24 h. The PZSE was calculated considering the pH value at which the curves crossed (pH = PZSE).

Statistical analysis

Pearson's correlation coefficient was determined to verify the degree of association between variables ($p < 0.01$ and $p < 0.05$). The PZSE and estimated PZC data were analyzed by the Student t-test ($p < 0.05$) in order to compare the means.

Results and Discussion

Soil physical characteristics

The studied soils differed substantially as regards texture even within the same soil class (Table 2). For clay content, Oxisols (P1 to P12) had the highest average value, then Ultisols (P13 to P18), Inceptisols (P19 to P22) and Entisols (P23). Higher clay content was found in P2 and in the subsurface diagnostic horizons of the P6 (Table 2). These results are in agreement with those found by other Brazilian authors (Matos et al., 2001; Mello et al., 2006; Pacheco et al., 2018).

The clayey soils showed a predominance of clay minerals with a majority of pH-dependent charges, which enhances P adsorption, especially at low pH values. Phosphate adsorption capacity has been shown to be dependent on the variation in the Fe oxide mineralogy of the clay fraction (Fontes and Weed, 1996).

The silt/clay ratio and WDC were inversely related to the degree of soil weathering, i.e. the higher the silt/clay ratio and WDC, the less the pedogenesis (Resende et al., 2007). A direct relationship was observed with DF since the higher this variable is, the greater the pedogenesis. In general, the Oxisols had a lower silt/clay ratio, lower WDC, and higher DF, followed by Ultisols and Inceptisols (Table 2). The silt/clay ratio of the Ultisols (0.57) was close to the average value found for the Oxisols (0.40), showing that soils of both classes are at an advanced weathering stage.

In the soils, the silt + clay ratio was higher in Inceptisols, followed by Oxisols and Ultisols (Table 2). In the Oxisols, the lower contents of silt + clay found in P3 and P10 are due to the parent material being derived from granitic rock (Ministério das Minas e Energia, 1982), as observed in the collection sites located in Itacarambi (P3) and Prata (P10), while the highest

Table 2 – Physical characteristics of the studied soils.

Soil Id.	Horizon	Coarse sand	Fine sand	Silt	Clay	WDC	DF	Silt	Textural class
		dag kg ⁻¹						%	Clay
P1	A	13	10	13	64	16	75	0.20	Very clayey
P1	Bw	12	9	11	68	13	81	0.16	Very clayey
P2	A	3	4	13	80	8	90	0.16	Very clayey
P2	Bw	2	3	12	83	13	84	0.14	Very clayey
P3	A	19	57	13	11	4	64	1.18	Sandy loam
P3	Bw	13	50	10	27	5	81	0.37	Sandy clay loam
P4	A	34	8	10	48	15	69	0.20	Clay
P4	Bw	25	6	7	62	22	65	0.11	Very clayey
P5	A	17	9	18	56	14	75	0.32	Clay
P5	Bw	14	7	11	68	21	69	0.16	Very clayey
P6	A	7	5	23	65	6	91	0.35	Very clayey
P6	Bw	3	3	11	83	12	86	0.13	Very clayey
P7	A	5	37	29	29	6	79	1.00	Clay loam
P7	Bw	4	32	24	40	6	85	0.60	Clay
P8	A	14	41	15	30	7	77	0.50	Sandy clay loam
P8	Bw	10	36	12	42	8	81	0.28	Sandy clay
P9	A	15	10	33	42	12	71	0.78	Clay
P9	Bw	14	11	23	52	14	73	0.63	Clay
P10	A	27	55	7	14	nd	nd	0.50	Sandy loam
P10	Bw	20	57	6	17	nd	nd	0.35	Sandy loam
P11	A	14	41	15	30	7	77	0.50	Sandy clay loam
P11	Bw	10	36	12	42	8	81	0.28	Sandy clay
P12	A	9	6	21	64	nd	nd	0.33	Very clayey
P12	Bw	7	4	20	69	nd	nd	0.29	Very clayey
P13	A	30	10	23	37	16	57	0.62	Clay loam
P13	Bt	20	7	17	56	28	50	0.30	Clay
P14	A	19	34	25	22	2	91	1.14	Sandy clay loam
P14	Bt	20	28	12	40	9	78	0.30	Sandy clay
P15	A	32	22	23	23	nd	nd	1.00	Sandy clay loam
P15	Bt	35	24	8	33	nd	nd	0.24	Sandy clay loam
P16	A	8	7	51	34	17	50	1.50	Silty clay loam
P16	Bt	3	3	44	50	23	54	0.88	Silty clay
P17	A	23	20	14	43	7	84	0.33	Clay
P17	Bt	15	11	8	66	10	85	0.12	Very clayey
P18	A	48	20	7	25	nd	nd	0.28	Sandy clay loam
P18	Bt	32	19	5	44	nd	nd	0.11	Sandy clay
P19	A	2	2	74	22	20	9	3.36	Silt loam
P19	Bi	3	2	58	37	19	49	1.56	Silty clay loam
P20	A	29	31	23	17	10	41	1.35	Sandy loam
P20	Bi	28	11	42	19	12	37	2.21	Loam
P21	A	8	11	48	33	21	36	1.45	Silty clay loam
P21	Bi	5	6	47	42	17	60	1.11	Silty clay
P22	A	10	3	52	35	13	63	1.48	Silty clay loam
P22	Bi	9	3	49	39	18	54	1.25	Silty clay loam
P23	A	51	37	5	7	nd	nd	0.71	Loamy sand

WDC = water-dispersed clay; DF = degree of flocculation; nd = not determined.

contents (in excess of 90 %) were found in an Oxisol (P2) derived from mafic/ultramafic rocks of the Macaúbas Group (Ministério das Minas e Energia, 1982), located in Capão, MG. In the Ultisols, the silt + clay contents indicate a higher participation of finer particles in the diagnostic horizon.

For the Inceptisols, results of silt + clay presented maximum contents of 96 %, with a large participation of the first fraction, even in the diagnostic horizon. High WDC contents, associated with a low DF and high silt/clay ratio (Table 2), suggest a lower pedogenesis and water percolation capacity in the Inceptisols, due to the discontinuity of the porous system related to coarser texture and the mineralogy of the clay fraction, which is predominantly kaolinitic and micaceous. According to Mello et al. (2006), the Inceptisols are subject to erosion, leading to the eventual translocation of particles to lowlands and streams where ferric oxy-hydroxides may be reduced and release toxic metals, such as arsenic, into the environment.

In the P20, unlike the other Inceptisols, silt and clay fractions together reached a percentage of 40 % (Table 2) since the greater part of the parent material is from sandstone rocks belonging to the Araxá Group. Soils developed from this geology are usually gravelly, sometimes stony, with a medium texture and low-activity clay (Ministério das Minas e Energia, 1982). In this profile, it is possible that the highest clay contents found in the Bi horizon were favored by the coarser texture of the parent material, with an eventual illuviation of fine materials, which, however, does not allow for classifying it as a Bt horizon (Embrapa, 2013).

P22 corresponds to a soil derived from calcareous rocks from the Lagoa do Jacaré Formation (Bambuú Group) (Ministério das Minas e Energia, 1982) and has a finer grain size compared to P20. The silt/clay ratio is of the order of 1.6 (Table 2), reflecting the parent material and a low weathering of these soils possibly due to higher material resistance and lower water infiltration capacity. In the region of Montes Claros, in the north of Minas Gerais, where the profile P21 was collected, there is a great influence of the pelitic material of the Bambuí Group, with soils tending to present characteristics related to the sub-horizontality of the parent material.

The Entisols (P23) presented 7 % clay and 5 % silt (Table 2), indicating a large participation of coarse and fine sand in the soil and presenting as main negative influences low water retention capacity and nutritional reserve for plants.

Soil chemical characteristics

In the studied soils, except for the B horizon in P11, the values of pH in water were higher compared to those observed for the pH in KCl, indicating a balance of negative charges on the surface of soil colloids (Table 3). The balance of charges has been commonly used as an indicator of the soil net charge (Mekaru and Uehara, 1972).

Most of the profiles from P1 to P5, taken from the Oxisols, are dystrophic and acid, except for P3, which presents a eutrophic profile (Table 3). This Eustrtox is located near Itacarambi, suggesting the participation of a carbonate material from the Sete Lagoas Formation (Bambuú Group) (Ministério das Minas e Energia, 1982).

except P5. However, exchangeable acidity lower than $4 \text{ cmol}_c \text{ dm}^{-3}$ is not enough to classify a soil given the aluminum criterion (Embrapa, 2013). These results are in agreement with Simas et al. (2005).

In relation to pH, the soils presented acidity from very high ($\text{pH H}_2\text{O} \leq 5$) to low alkalinity ($\text{pH H}_2\text{O}$ between 7.1 and 7.8), according to Ribeiro et al. (1999a). The highest pH value corresponds to the surface horizon of P14, which is probably due to the agricultural management. Acidic soils can support high-level agriculture when correctly managed, as was evident in a number of regions in southern Minas (Fontes and Alleoni, 2006).

The TOC content (Table 3) in the surface layer also ranged from low (between 0.41 and 1.16 dag kg^{-1}) to very high ($> 4.06 \text{ dag kg}^{-1}$), according to Ribeiro et al. (1999a). On the surface of organic matter colloids the variable charge is negative, below pH values commonly found in humid tropical soils, capable of attracting cationic species, increasing the cation exchangeable capacity (CEC) of soils with a high content of organic matter (Fontes and Alleoni, 2006).

The P contents were low or very low (Ribeiro et al., 1999b) in all the Oxisol profiles (Table 3). In these soils, P adsorption reflects soil mineralogy. Crystalline Fe oxides are important adsorbents of P, with goethite apparently adsorbing more than hematite (Fontes and Weed, 1996). According to these authors, gibbsite also plays a key role in adsorption, mainly related to

mineral abundance. In this regard, the Oxisols P6 and P11 presented more pronounced goethite, hematite, and gibbsite (Table 4), and consequently, showed lower contents of P-rem in relation to the other studied soils. Similarly, P12 presented very low P-rem contents due to the goethite mineralogy, with a significant presence of gibbsite, in addition to its very clayey texture. Remanent phosphorus is inversely related to the phosphate adsorption capacity by soils. In other words, low P-rem values mean that the soils have a high capacity for adsorbing P and vice versa.

Another aspect involving the Oxisols P6 to P11 was the high affinity between micronutrients and Fe and Mn oxides. In general, the Fe and Mn contents were high, while Cu and Zn contents were considered higher (Ribeiro et al., 1999b) in profiles P7 and P14, respectively. These two elements tend to concentrate in soils derived from basic igneous rocks, while B tends to predominate in sedimentary rocks (Krauskopf, 1972) which explains the very low contents of this element.

Higher values for P-rem (Table 3), such as those presented by P23, followed by the Ultisols (P13 to P18), except for the very clayey horizon of P15, and Inceptisols (P19 to P22) are associated with more kaolinitic and less oxidic soils (Table 4) (Schaefer et al., 2008).

For the Ultisols, variable values of base saturation and Al saturation occur because part of these soils are eutrophic (P14 and P17) and the others (P13, P15, P16, and PV18) are dystrophic or alic (Table 3), according to

Table 4 – Mineralogical composition of the studied soil.

Soil Identification	Sand	Silt	Clay
P1	Kt, Gb, Qz, Mt/Mh, Hm	Kt, Qz, Mi	Kt, Gb, Gt, Il, Hm
P2	Qz, Pg	Kt, Gb, Qz, Gt, Mi	Il, Kt, Gb, Hm, Gt
P3	Qz, Pg, Hm	Kt, Gb, Qz, Gt, Mi	Il, Kt, Gt, Qz, Hm
P4	Qz, Pg	Kt, Gb, Qz	Kt, Gb, Gt
P5	Kt, Gb, Qz, Pg, Hm	Kt, Gb, Gt, Qz	Kt, Gb, Gt, Hm
P6	Kt, Pg, Gb, Mi, Felds-K, Qz	Mi, Kt, Gb, Qz, Felds-K, Pg	Il, Kt, Gb, Gt
P7	Kt, Gb, Qz, Mt/Mh, Hm	Kt, Gb, Gt, An, Qz, Mh, Hm	Kt, Gb, Gt, Hm, Il
P8	Qz, Pg	Mi, Kt, Gb, Qz, Felds-K	HIV, Il, Kt, Gb, Gt, Mh Hm
P9	Qz, Pg	Kt, Qz, Pg, Hm	Il, Kt, Gt, Hm
P10	Qz, Pg, Hm	Qz	HIV, Il, Kt, Gb, Gt, Hm
P11	Qz, Pg	Qz, Hm	Il, Kt, Gb, Gt, Hm
P12	Kt, Gb, Qz, Pg, Hm	Mh, Kt, Gb, Qz, An, Hm	Kt, Gb, Gt, Il, Mh, Hm
P13	Qz, Pg, Felds-K	Mi, Kt, Qz, Pg, Felds-K	Il, Kt, Gb, Gt, Hm
P14	Mi, Qz, Pg	Kt, Mi, Qz, Pg	Il, Kt, Gb, Gt, Hm
P15	Qz, Pg	Kt, Qz	Il, Kt, Gt, Hm
P16	Qz, Pg	Mi, Kt, Qz, Pg, Felds-K	Il, Kt, Gt, Hm
P17	Qz, Pg	Kt, Mi, Qz, Pg	Kt, Gt, Il
P18	Qz, Pg	Kt, Qz	Il, Kt, Gt, Hm
P19	Mi, Qz, Gt, Pg	Mi, Kt, Qz, Pg, Felds-K	Il, Kt, Gt, Felds-K, Hm
P20	Mi, Qz, Pg, Felds-K	Mi, Kt, Qz, Pg, Felds-K	Vm/HIV, Il, Kt
P21	Qz, Pg, Felds-K, Mi	Mi, Kt, Qz	Il, Kt, Gt, Felds-K, Hm
P22	Mi, Qz, Felds-K, Pg	Mi, Qz, Kt	Il, Kt, Qz
P23	Qz, Pg	Kt, Qz	Kt, Qz

An = anatase; Kt = kaolinite; Felds-K = potassium feldspar; Gb = gibbsite; Gt = goethite; Hm = hematite; Il = illite; Mi = mica; Mt/Mg = magnetite/maghemite; Mh = maghemite; Qz = quartz; Pg = plagioclase; Vm/HIV = vermiculite/hydroxy-interlayered vermiculite; HIV = hydroxy-interlayered vermiculite.

Embrapa (2013). However, one of the most pronounced characteristics is related to the micronutrient content in the superficial horizons of these soils, which are among the highest of all the studied soils. Nutrient cycling in the A horizon, as well as the affinity of certain elements for organic matter, may possibly be responsible for this accumulation.

In eutrophic soils, the contents of Ca^{2+} and Mg^{2+} are also high in all profiles, especially in the superficial horizon. Even in dystrophic Ultisols, it is common for the surface horizon to have high base saturation, giving an epi-eutrophic character to these soils (P13 and P16) (Embrapa, 2013). This influences soil pH values, which tend to be higher than in the Oxisols, especially in P14, which is classified as having low alkalinity according to Ribeiro et al. (1999a).

The Inceptisols presented low to high acidity (Ribeiro et al., 1999b) with an average pH value for the profiles of approximately 5.5 (Table 3). These values are above the estimated soil PZC values (Table 5), with a greater predominance of negative charges in the pH range when compared to other soil classes, which is probably due to the higher presence of minerals with negative charges resulting from isomorphic substitution (IS), such as micas. The high contents of P-rem in these soils may be related, among other factors, to this existing negative charge balance, a high amount of silt and the lower relative amount of Fe oxides, and the virtual absence of gibbsite.

The set of physical, chemical and mineralogical characteristics gives the soil a specific capacity to retain anionic species. The Inceptisols, due to their lower Fe and Al content, especially gibbsite, were the soil class with the lowest adsorption of anionic species (Almeida et al., 2021).

The Inceptisols, represented by the profiles P19, P20 and P21, presented high Al^{3+} contents (Table 3), mainly P20, whose values were higher than $4 \text{ cmol}_c \text{ dm}^{-3}$ and Al saturation higher than 50 % were sufficient to be classified as having an Al character (Embrapa, 2013). High Al contents are not always due to intense soil weathering, but to the pelitic material i.e., the low contents of bases and the presence of Al in the mica and feldspar structure causes young soils, developed from pelitic rocks, to have high Al^{3+} contents whereas, in older soils, Al contents in solution tend to be lower due to the formation of gibbsite (Resende et al., 2007). Thus, these soils presented a low sum of bases, with Ca^{2+} and Mg^{2+} contents considered very low/medium (Ribeiro et al., 1999b) in all soil profiles.

On the other hand, high K⁺ contents were found. This element is considered the most common in soils derived from pelitic rocks, which is a consequence of the micaceous mineralogy and probably of a higher substitution of Al by Si, allowing for K maintenance in the structure of micas (Resende et al., 2007). This fact is evidenced by the appearance of mica in the silt fraction of all the studied soils and feldspar in most of them

Table 5 – Mineralogical and electrochemical characteristics of the studied soils and Fe contents obtained from the clay fraction by three successive extractions of dithionite-citrate (Fed) and a single extraction of ammonium oxalate (Feo).

Soil Identification	Horizon	Gibbsite Kaolinite		Fed		Fe _o		ZPC	ZPSE
		— % —	— dag kg ⁻¹ —	Fed	Feo				
P1	A	24.4	38.6	7.43	0.10	0.01	3.64	3.64	
P1	Bw	22.6	48.9	4.16	0.59	0.14	4.68	4.89	
P2	A	41.9	24.0	6.34	0.13	0.02	3.56	3.85	
P2	Bw	40.3	26.0	5.41	0.73	0.13	4.65	4.99	
P3	A	5.7	62.4	3.46	0.29	0.08	4.39	4.91	
P3	Bw	2.9	62.6	2.95	1.45	0.49	3.39	3.42	
P4	A	2.2	85.3	1.74	0.11	0.06	2.96	3.25	
P4	Bw	1.4	78.5	1.61	1.20	0.74	2.89	4.96	
P5	A	6.5	70.4	5.55	1.31	0.23	3.52	4.55	
P5	Bw	6.0	68.4	5.70	1.14	0.20	4.53	4.60	
P6	A	15.2	40.9	2.47	0.18	0.07	3.61	3.99	
P6	Bw	12.2	46.4	1.74	0.98	0.56	3.87	3.73	
P7	A	nd	nd	4.09	2.02	0.49	3.05	3.52	
P7	Bw	25.0	49.3	5.42	3.22	0.59	3.26	3.76	
P8	A	26.0	35.1	5.03	0.17	0.03	4.81	4.25	
P8	Bw	23.8	31.2	4.27	0.99	0.23	3.87	4.42	
P9	A	6.0	70.1	7.37	0.19	0.02	5.89	5.13	
P9	Bw	0.0	66.7	6.31	1.25	0.19	3.92	4.56	
P10	A	11.1	32.4	5.61	1.99	0.35	2.79	3.57	
P10	Bw	15.2	26.4	10.38	2.03	0.19	3.28	5.37	
P11	A	10.1	39.8	9.69	0.56	0.05	2.75	3.50	
P11	Bw	9.4	36.8	10.47	0.44	0.04	5.91	3.54	
P12	A	32.7	29.3	6.37	1.96	0.30	3.09	4.03	
P12	Bw	32.9	24.1	8.48	1.12	0.13	2.95	4.94	
P13	A	6.9	62.3	4.57	0.25	0.05	3.43	3.22	
P13	Bw	nd	nd	4.22	0.95	0.22	3.61	4.43	
P14	A	15.6	40.8	4.83	0.47	0.09	5.73	5.98	
P14	Bw	7.9	33.8	5.41	2.34	0.43	3.64	3.37	
P15	A	0.7	67.7	1.86	0.14	0.07	4.36	4.66	
P15	Bw	2.1	66.0	0.75	0.19	0.25	4.06	3.34	
P16	A	6.7	44.2	4.08	0.31	0.07	3.75	3.64	
P16	Bw	3.0	32.2	3.38	2.12	0.05	3.22	5.32	
P17	A	4.2	67.7	6.05	0.26	0.04	3.99	4.34	
P17	Bw	2.0	70.4	6.01	0.85	0.14	4.89	2.86	
P18	A	3.0	80.8	1.52	0.21	0.13	3.23	3.16	
P18	Bw	1.3	79.3	1.34	1.00	0.74	3.12	2.98	
P19	A	19.9	68.6	6.37	0.28	0.04	3.04	3.48	
P19	Bi	7.0	34.6	4.66	4.95	1.06	2.73	2.98	
P20	A	5.7	56.5	1.07	0.23	0.21	2.71	3.08	
P20	Bi	4.0	59.7	0.86	1.29	1.50	2.52	2.92	
P21	A	nd	nd	6.39	0.22	0.03	2.22	2.72	
P21	Bi	0.9	34.3	3.97	1.57	0.39	2.91	3.44	
P22	A	0.7	18.3	3.38	0.23	0.06	4.39	4.58	
P22	Bi	2.0	17.4	3.20	1.70	0.53	4.12	4.44	
P23	A	1.6	80.6	0.36	0.16	0.44	3.21	nd	

ZPC = Zero Point of Charge; ZPSE = Zero Point of Saline Effect; nd = not determined.

(Table 4). P2 was the only profile to present high base saturation, with an average of 84 % (Table 3). This is due to the high exchangeable Mg^{2+} and Ca^{2+} contents from a

parent material of sandstone rocks of the Araxá Group, as previously discussed. In general, when considering the macroelements, the Inceptisols are rich in K only, and poor in Ca, Mg, and P. The contents of P and S were very low (Table 3), according to Ribeiro et al. (1999a).

As regards the micronutrients (Table 3), Mn and Fe presented extremely high available contents (Ribeiro et al., 1999b), mainly in P19, due to the ferriferous parent material. The presence of Fe oxides in the soil is essential since they practically control the solubility of this element, which is very much influenced by pH and soil redox potential (Krauskopf, 1972).

In profile P22, the available Fe contents were low, in fact in the literature they are considered very low (Ribeiro et al., 1999b) an observation corroborated by the absence of Fe oxides (Table 4). Soils derived from pelitic rocks tend to present higher B contents, as they are more concentrated in sediments of marine origin (Resende et al., 2007). However, this trend could not be observed since the B contents were in general low or very low in all profiles.

The chemical characteristics of Entisols (P23) involve very low values for both micro and macronutrients, with low base saturation and high Al saturation although the exchangeable and potential acidity contents are also low (Table 3). The acidity of these soils is considered as high (Ribeiro et al., 1999b) and is possibly related to the quartz weathering present in their sand, silt, and clay fractions (Table 4), which release H_4SiO_4 (Resende et al., 2007). Due to the sandy texture and low CEC, Entisols exhibit lower retention capacity of inorganic contaminants as compared to Oxisols (Melo et al., 2011).

Mineralogy

The soil mineralogy results (Table 4) indicate the presence of kaolinite, gibbsite, goethite, illite, hematite, hydroxy-interlayered vermiculite, and maghemite, among others. The presence of minerals was confirmed in the sand, silt and clay fraction via interplanar spacing (d). These minerals are in agreement with those observed by other authors for Brazilian soils (Fontes and Weed, 1991; Fontes and Carvalho Jr., 2005; Simas et al., 2005; Schaefer et al., 2008; Camêlo et al., 2017; Camêlo et al., 2018; Pacheco et al., 2018).

Goethite seems to be the most stable and abundant form of Fe oxide in tropical soils. The factors favoring its formation, to the detriment of hematite, are lower temperatures, higher H_2O activity, high organic matter content, and lower Fe content in the parent material (Schwertmann and Taylor, 1989). The association between goethite and hematite, a common characteristic in most soils in tropical regions, was very well evidenced in these soils (Schwertmann, 1985).

In the most weathered and acidic soils, gibbsite is observed in large quantities, especially in the Oxisols P2 and P12 (Tables 4 and 5). Because of the relatively high soil development, the tendency for the Ultisols was

similar for the Fe and Al oxy-hydroxides, in addition to the common presence of kaolinite and illite. However, the absence of significant amounts of gibbsite, which were detected only in thermal analysis (Table 5), shows that the Ultisols, in general, are less developed in relation to the Oxisols, which is in agreement with the theory of soil formation.

According to Schaeffer et al. (2008) relatively large contents of gibbsite tend to occur in Brazilian Oxisols, where weathering and leaching processes are more intense. According to these authors, the higher presence of gibbsite, together with goethite, may favor the formation of aluminized goethite, which tends to occur mainly in tropical climate soils with a well-defined dry and humid season. These climate characteristics are found in most of the state of Minas Gerais.

The presence of illite in all samples of the clay fraction and mica in all samples of the silt and sand fractions stood out in the Inceptisols (Table 4). Illite interplanar spacing (1.00 nm) has the characteristic of not changing in treatments with Mg or K, with or without heating (Whittig and Allardice, 1986).

The P7 and P9 profiles presented in their mineralogical constitution hydroxy-interlayered vermiculite (HIV) and P20 presented vermiculite (Vm) in addition to HIV. Interplanar spacing (d) 1.43 nm (HIV) and 1.0 nm (mica/illite) were similar in the samples taken of Fe oxides and in those with the treatment of K at ambient temperature. When heated to 550 °C, the entire HIV has its Al polymers destroyed and collapses at a (d) of 1.0 nm (Whittig and Allardice, 1986). However, in the profile P20, the 1.0 nm has already undergone an increase in intensity in the treatment with K at ambient temperature, indicating that part of the material is composed of Vm and another part remains at 1.43 nm, which is HIV. At 550 °C, all the material collapses at 1.0 nm on the destruction of the interlayer material.

The crystalline forms of Fe associated with soil clay fraction (Fe_d), quantified by dithionite-citrate analysis (Coffin, 1963), and those with lower crystallinity (Fe_o), obtained from a single extraction with ammonium oxalate (McKeague and Day, 1966) are shown in Table 5. The contents of Fe_2O_3 , extracted by dithionite (Fe_d), were variable. The highest values (around 10 $dag\ kg^{-1}$) were found in two Oxisols (P9 and L10). On the other hand, high values obtained from the Fe-oxalate (Fe_o) extraction, corresponded to profile P6.

The ratio Fe_o/Fe_d can be a proxy for the soil developed degree, being smaller in weathering soils (Cornell and Schwertmann, 2003). In general, the values of Fe_o/Fe_d showed a predominance of well crystallized Fe forms for the Ultisols and Oxisols, and poorly crystallized Entisols and Inceptisols (Table 4). The Fe_o/Fe_d ratios were higher in the Bi horizon of two Inceptisols (P19 and P20). These results were also in agreement with those reported by Pacheco et al. (2018), in which the authors found a predominance of well crystallized Fe in more weathered soils, such as

Oxisols. Highly weathered tropical soils have very low amounts of poorly crystallized Fe oxides (Fontes and Weed, 1991).

The literature highlights the effect of organic matter on the inhibition of Fe crystallization, due to the strong complexation of poorly crystallized Fe oxides by organic compounds (Schwertmann, 1966; Simas et al., 2005). However, for most of the studied soils, a lower proportion of poorly crystallized material was found in the surface horizon, demonstrating little influence of the organic matter on the increased Fe_o .

The presence of silica in the system inhibits the formation of Fe oxides with higher crystallinity (Schwertmann and Taylor, 1989). For this reason, the profile P4, which presented high contents of kaolinite (78 % in the Bw horizon), also presented the highest Fe_o/Fe_d ratio in the subsurface horizon (Table 5).

Point of zero charge of soils

The results show high correlation between PZC and the exchangeable Al contents and the Fe_o/Fe_d ratio, both in the A and B horizons of the soil classes (Table 6).

The negative correlation found between exchangeable Al and PZC can be of great importance as regards management practices in soil classes with very low PZC values (consequently high exchangeable Al). Simas et al. (2005) reported that liming is not recommended when the Al^{3+} content in soils requires extremely high $CaCO_3$ doses, since the practice may not be economically viable or may accelerate the mineralization of organic compounds.

An examination of the relationship between Fe_o/Fe_d and the PZC of the evaluated soil classes in this study, reaffirms the role of mineralogy in the PZC of soils. In highly weathered soils in the state of São Paulo (Alleoni and Camargo, 1994), PZC showed a positive correlation with gibbsite and pH. Negative correlation between PZC and the soil weathering index was also observed by these authors. Since the ratio of Fe_o/Fe_d can be used to estimate the soil weathering degree (Cornell and Schwertmann, 2003), the negative correlation observed between PZC and Fe_o/Fe_d in this study corroborates the result observed by Alleoni and Camargo (1994). However, in the main classes of soils in the state of Minas Gerais, the correlation between PZC and gibbsite was not observed.

Although the effect of organic matter on PZSE is known in the literature (Hendershot and Lavkulich, 1979) to cause its decrease, a negative statistical correlation was not established in this study probably due to the differentiation between the organic matter from distinct localities and vegetation cover, conferring diverse effects and a direct relationship is less evident.

A comparison between both methods (PZC and PZSE) was undertaken, which showed positive correlation between them in the A horizon ($r = 0.89$) while in the B horizon ($r = 0.04$) correlation was not significant at $p < 0.01$. The t-test (Table 7) did not

Table 6 – Correlation between physical, chemical and mineralogical attributes and the ZPSE and estimated ZPC of A and B horizons of the studied soils.

Attributes	A horizon		B horizon	
	ZPSE	ZPC	ZPSE	ZPSE
T	ns	ns	ns	ns
Al	ns	-0.686*	ns	-0.689*
H+Al	ns	ns	ns	ns
SB	ns	ns	ns	ns
V (%)	ns	ns	ns	ns
m (%)	ns	-0.464**	ns	-0.469**
TOC	ns	ns	ns	ns
Clay	ns	ns	ns	ns
Fed	ns	ns	ns	ns
Feo	ns	-0.487**	ns	-0.512**
Feo/Fed	-0.484**	-0.597*	-0.525**	-0.619*
pH	ns	ns	ns	ns
P-rem	ns	ns	ns	ns
Gibbsite (Gt)	ns	ns	ns	ns
Kaolinite (Kt)	ns	ns	ns	ns
Gb+Kt	ns	ns	ns	ns
Gb/(Gb+Kt)	ns	ns	0.431**	ns

ns, ** and * = Not significant, significance level $\alpha = 0.01$ and significance level $\alpha = 0.05$, respectively; T = total cation exchange capacity; SB = sum of base; V (%) = base saturation; m (%) = Al^{3+} saturation; TOC = total organic carbon; Prem = phosphorus remaining; Fed = dithionite-citrate; Feo = Fe ammonium oxalate; ZPC = Zero Point of Charge and ZPSE = Zero Point Saline Effect.

Table 7 – Comparison between ZPSE and estimated ZPC of A and B horizons of the studied soils.

	A horizon		B horizon	
	ZPSE	ZPC	ZPSE	ZPC
Mean	3.95 ± 0.78 a	3.67 ± 0.94 a	4.07 ± 0.81 a	3.72 ± 0.82 a
Minimum	2.72	2.22	2.86	2.52
Maximum	5.98	5.89	5.37	5.9
Median	3.72	3.53	4.09	3.625

ZPSE = Zero point of Saline Effect; ZPC = Zero Point of Charge; Means followed by the same letter do not differ from each other by the Student t-test ($p < 0.05$).

indicate a significant difference between methods, as well as between soil classes and their A and B horizons. A similar analysis was performed in relation to soil classes and no differentiation was observed between them.

The importance is related to the proposition of methodologies for other studies. Due to the degree of simplification that the equation of Keng and Uehara (1974) provides, its use should be considered when the focus of the study does not need a higher degree of refinement.

Among the soil classes, the results showed that the Inceptisols presented the lowest PZC values (Table 4), denoting their incipient character and higher density of negative charges, while more oxidic soils presented the highest PZC values (up to a maximum of 5.9).

Most of the studied soils presented pH values (Table 3) higher than the ZPZC values (Table 5), evidencing their electronegative character despite being highly weathered and oxidic. The only exception is the B horizon of profile P11, whose pH in water was higher than its PZC, thus presenting an electropositive character. Once the $\text{pH} < \text{PZC}$, the surface of the colloids is positively charged, favoring adsorption of the anionic species (Fontes, 2012).

Conclusions

The evaluated soils of Minas Gerais differed substantially in terms of texture and fertility, even within the same soil class. Soils presented textures ranging from loamy sand to very clayey with profiles varying from dystrophic to eutrophic, which suggests the participation of the parent material.

Most of these soils presented an advanced stage of weathering with a typical mineralogical composition of highly weathered soils composed of kaolinite, gibbsite, goethite, hematite and illite. They also showed traces of hydroxy-interlayered vermiculite and maghemite.

A positive correlation was observed between the point of zero salt effect (PZSE) and the equation developed by Keng and Uehara (1974) for estimating the point of zero charge (PZC) of soils in the A horizon.

Significantly high negative correlation ($p < 0.01$) was observed between PZC and the exchangeable Al contents and the Fe_o/Fe_d ratio, both in the A and B horizons of the soil classes, evidencing the influence of chemicals and mineralogy on the PZC of soils.

Due to the diversity of soils in the state of Minas Gerais, the results obtained reinforce the importance of very detailed descriptions of soil attributes with a view to improving both usage and soil management.

Acknowledgments

The authors are grateful to the Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brasil (CNPq) for the financial support. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Authors' Contributions

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