

Division - Soil in Space and Time | Commission - Soil Genesis and Morphology

# Reversibility of the Hardening Process of Plinthite and Petroplinthite in Soils of the Araguaia River Floodplain under Different Treatments

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**ABSTRACT:** Although ferruginous materials occur frequently in soils of tropical regions, information about the reversal of the hardening process of these materials is scarce. This study assessed the influence of different chemical treatments and periods of immersion on the reversibility of the hardening process of plinthite and petroplinthite in soils of the Araguaia River plain. Soil samples were collected from the plinthic horizons in 0.10 m high and 0.15 m diameter PVC cylinders and divided into subsamples with a rock hammer. Homogeneous petroplinthite samples were also collected and broken into subsamples with a rock hammer. The plinthite and petroplinthite subsamples were subjected to five immersion treatments: distilled water, calcium carbonate solution, sodium hydroxide solution, sodium hydroxide solution + sodium hexametaphosphate, and acidic solution. The subsamples were immersed for 20, 40, 80, and 160 days. The dispersion and stability degrees and compressive strength in these subsamples were assessed. The wet aggregate stability test indicated no impact on the structural stability of plinthite and petroplinthite subsamples subjected to the treatment with different chemical agents, demonstrating the non-reversibility of the hardening process of these materials of the Araguaia River floodplain, under the studied conditions.

**Keywords:** dispersion, deterioration of structural stability, hardness, reagents, iron oxides.

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## INTRODUCTION

Plinthite occurs in soils of flat or slightly sloped areas, rich in iron, and exposed to seasonal variations of the groundwater level. It is the result of subsurface accumulation of iron hydroxides, kaolinite, and quartz, which may undergo irreversible hardening and consequently be transformed into petroplinthite (Daniels et al., 1978; Gardi et al., 2014). Thus, petroplinthite is an extremely consolidated and firm material when moist and extremely hard when dry (Alexander and Cady, 1962; Santos et al., 2013).

The progressive hardening of ferruginous materials is related to their higher iron content that results in increased crystallinity (Santos and Batista, 1996). Soil iron can originate from other horizons, higher sites or result from the elimination of silicon cations and base ions through leaching of compounds dissolved by weathering (Gardi et al., 2014; Soil Survey Staff, 2014). Iron is transported by water under anaerobic conditions as ferrous ion and precipitates in soft clayey concretions of iron oxides that harden after drying (Gardi et al., 2014).

Ferruginous crusts containing only Fe and Al oxides do not provide plants with nutrients and have low water retention capacity (Alexander and Cady, 1962). On the other hand, ferruginous crusts with kaolinite contain water and channels that can be penetrated by roots. Some laterites contain encapsulated weathering minerals, such as micas and feldspars which, when ruptured, can provide nutrients. The presence of vegetation in lateritic areas is an important factor to reverse the hardening process of this material, which is caused by reduction and chelation with organic matter, by the mechanical action of roots, and mitigation of temperature and drought (Alexander and Cady, 1962).

Coelho et al. (2001) studied the development of nodular ferricretes (iron-rich crusts) in sandstone of the Bauru Group, and concluded that the cortex of the petroplinthic profiles was yellowish, suggesting dissolution and removal of hematite, favoring the presence of goethite. This fact characterizes the destruction of the ironstone layer and may be related to climate or lithological factors, indicating that these conditions contrast with paleoclimatic conditions that favor ferricrete formation, such as prolonged droughts or high iron contents.

Apart from some studies on plinthite, Plinthosols (WRB, 2014) and *Plintossolos* [Brazilian Soil Classification System (Santos et al., 2013)] and related issues (Daniels et al., 1978; Zhang and Karathanasis, 1997; Ogunwole et al., 2001; Eze et al., 2014), there is very scarce or no reliable information at all on the reversal of hardening of ferruginous materials, which occurs frequently in soils of tropical regions. This study addressed the impact of different chemical, mechanical, and immersion treatments on the reversibility of plinthite and petroplinthite hardening in soils of the Araguaia River floodplain, to contribute to a more accurate classification of these materials.

## MATERIALS AND METHODS

Plinthite and petroplinthite samples from diagnostic horizons of two profiles (P2 and P5, respectively) of soils of the Araguaia River floodplain and surroundings, in Luiz Alves, district of São Miguel do Araguaia, Goiás (Table 1) were used in the study. The profiles were selected based on field observations related to the occurrence of these materials in the soil, and were described according to the recommendations of the Manual of Field Soil Description and Sampling (Santos et al., 2015) and the Technical Manual of Pedology (IBGE, 2015). The studied soils were classified according to the Brazilian Soil Classification System (Santos et al., 2013) and World Reference Base for Soil Resources (WRB, 2015), to characterize each sampling point.

Regional climate is type AW, defined as tropical with dry winters, according to the Köppen-Geiger classification system. The average annual rainfall ranges between 1,800-2,000 mm and the average annual temperature between 25-26 °C (Cardoso et al.,

2014). Profile P2 lies on the floodplain and possibly on paleo-channels, owing to its physical nature (mean texture) (Table 2), while P5 lies outside the floodplain, approximately 6.40 km away from Luiz Alves, towards São Miguel do Araguaia, in a higher adjacent area with good natural drainage.

From profile P2, due to its plinthic horizon, undisturbed samples were collected in PVC cylinders (height 0.10 m, diameter 0.15 m), inserted directly into the plinthic horizons (4Btfc2 and 4Btfc3, to a depth between 0.61 and 1.10 m), with three replicates. Afterwards, in a protected environment, plinthite subsamples were randomly extracted by hand from the collected samples, according to visual (color) and physical or mechanical (consistency) criteria (IBGE, 2015). After division, the consistency of most plinthite samples ranged from soft to hard (IBGE, 2015; Santos et al., 2015), when manually examined.

In profile P5, three homogeneous samples of petroplinthite were collected in the form of continuous blocks (lateritic concretion, “canga”), weighing approximately 400 g per sample. The most distinctive criterion was the great hardness (very hard consistency in dry sample). Petroplinthite subsamples with a mean volume of 7 cm<sup>3</sup> were collected from these profile samples in the laboratory, using a rock hammer.

The plinthite and petroplinthite subsamples of both profiles were individually weighed in semi-analytical scale, placed in plastic containers and subjected to five wetting treatments for 20, 40, 80, and 160 days, which simulate field situations, conditions promoted by use and soil management, and alterations that may occur in the laboratory, to investigate

**Table 1.** Location of the profiles and characterization of the study sites

Location	Altitude	Geology <sup>(1)</sup>	Original material <sup>(1)</sup>	Local relief	Current land use
m					
Profile 2 - <i>Plintossolo Argilúvico Eutrófico petroplíntico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)					
13° 10' S; 50° 31' W	220	Sedimentary cover of Bananal	Alluvial sediments	Flat	Soybean
Profile 5 - <i>Plintossolo Pétrico Concrecionário típico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)					
13° 15' S; 50° 31' W	225	Estrondo Group	Weathering of micaxist with grenade and sericite xist	Gently undulating	Natural vegetation

<sup>(1)</sup> Brasil (1981).

**Table 2.** Horizon layers, grain size composition, and textural class of the studied profiles

Horizon	Layer	CS	FS	Silt	Clay	TC
m						
g kg <sup>-1</sup>						
Profile 2 - <i>Plintossolo Argilúvico Eutrófico petroplíntico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)						
Ap1	0.00-0.06	29	281	321	369	CL
2Ap2	0.06-0.18	133	448	237	182	SL
3AE	0.18-0.27	175	455	229	141	SL
3EA	0.27-0.34	157	433	249	161	SL
3Efc	0.34-0.50	75	376	327	222	L
3Btfc1	0.50-0.61	71	354	312	263	CL
4Btfc2	0.61-0.76	91	358	329	222	L
4Btfc3	0.76-1.10 <sup>+</sup>	89	366	323	222	L
Profile 5 - <i>Plintossolo Pétrico Concrecionário típico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)						
Apc	0.00-0.09	154	395	167	284	SCL
Bc1	0.09-0.56	102	285	165	448	C
Bc2	0.56-1.03 <sup>+</sup>	138	297	118	447	C

Coarse sand, fine sand, silt, and clay determined by densimeter method (Donagema et al., 2011). CS = coarse sand; FS = fine sand; TC = textural class; CL = clay loam; SL = sandy loam; L = loam; SCL = sandy clay loam; C = clay.

the effect of the different time periods on these materials. The five treatments consisted of: DW - distilled water, simulating normal wetting of soils with plinthic materials caused by flooding or rainfall; CC - calcium carbonate solution (3.33 g L<sup>-1</sup> calcium carbonate), simulating a management with liming; SH - 10 % sodium hydroxide (40.00 g L<sup>-1</sup> sodium hydroxide) solution, simulating the application of conventional soil dispersants; SH + HM - 5 % sodium hydroxide solution + 5 % sodium hexametaphosphate (40.00 g L<sup>-1</sup> sodium hydroxide + 35.70 g L<sup>-1</sup> sodium hexametaphosphate), also simulating the application of conventional soil dispersants; AS - acidic solution (6.80 g L<sup>-1</sup> monopotassium phosphate), theoretically simulating a characteristic pH range of soils with plinthic horizons in Brazil, of around 5.0, for maintaining the buffering of the soil solution. The pH of the soils was determined in water and 1 mol L<sup>-1</sup> KCl, at a soil-liquid ratio of 1:2.5. During the study period, the pH of the solutions was measured weekly and the mean pH value calculated for each solution at the end of the assessment period (Table 3).

For each immersion treatment, the following properties were assessed sequentially in the subsamples: material dispersion degree, stability and compressive strength of plinthite and petroplinthite, with 10 replications (10 subsamples). Because of the great variability among the materials of the same horizon (highly irregular populations), a selective mean was used for assessment, after exclusion of 30 % of the most dispersed data from the general mean.

To analyze the degree of dispersion, plinthite and petroplinthite subsamples were removed from the solutions, transferred to aluminum containers and oven-dried at 105 °C. The subsamples were then weighed, and the percentage dispersion was calculated using the equation 1:

$$D = \left[ \left( \frac{W_0}{W_f} - 1 \right) \right] \times 100 \quad \text{Eq. 1}$$

in which: W<sub>0</sub> is the initial sample weight (g) and W<sub>f</sub> the final sample weight (g), corresponding to the material retained in 2-mm-diameter mesh.

Proportional stability degree of plinthite and petroplinthite subsamples was analyzed by wet sieving in a Yoder wet-sieving apparatus (Donagemma et al., 2011). The subsamples were individually weighed, pre-moistened by capillarity, placed on moist filter paper, transferred to two sets with five sieves (diameter 2.00; 1.00; 0.50; 0.25; and 0.106 mm) and subjected to vertical oscillation in water containers for 15 min (40 cycles min<sup>-1</sup>). The material retained in each sieve was transferred to aluminum containers and oven-dried at 105 °C. Subsequently, the weight and percentage of stable materials in each diameter class were calculated.

Compressive strength of plinthite and petroplinthite subsamples was assessed with a universal mechanical testing equipment (Instron®, model 3367, Grove City, United States) with a 30 kilonewton (kN) load cell. The maximum force, expressed in newtons

**Table 3.** pH values of the diagnostic horizons and mean pH of the immersion solutions of plinthite (PL) and petroplinthite (PP) subsamples from the studied soil profiles

Horizon	pH <sup>(1)</sup>		Material	pH solution				
	H <sub>2</sub> O	KCl		DW	CC	SH	SH + HM	AS
P2 - <i>Plintossolo Argilúvico Eutrófico petroplíntico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)								
4Btfc2	5.00	4.20	Plinthite	7.56	7.44	12.18	9.72	5.60
4Btfc3	5.20	4.20	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
P5 - <i>Plintossolo Pétrico Concrecionário típico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)								
Bc1	5.30	5.10	Petroplinthite	7.77	7.64	10.78	9.88	5.91
Bc2	5.40	5.80	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

<sup>(1)</sup> pH(H<sub>2</sub>O) and pH(KCl) at a soil-liquid ratio of 1:2.5. DW = distilled water; CC = calcium carbonate; SH = sodium hydroxide; SH + HM = sodium hydroxide and sodium hexametaphosphate; AS = acid solution; n.a. = not analyzed.

(N), was reached when the equipment deformed constantly 40 % compared to the initial height of the material, at a speed of  $0.50 \text{ mm s}^{-1}$ . Due to the irregular shapes, the plinthite and petroplinthite subsamples were measured with a digital caliper, taking three measurements (height, width, and length) to calculate the approximate volume of each sample. Compression test values were expressed in  $\text{N cm}^{-3}$ .

Qualitative and quantitative analyses of the chemical composition of representative samples of plinthite and petroplinthite were determined by X-ray fluorescence spectrometry (XRF) to facilitate the characterization of the material. The individual samples were pulverized in an agate mortar and pelleted, and then compacted for 1 min in a 5 ton hydraulic press. The pressed pellets were analyzed in an EDX-700 HS Energy Dispersive X-ray Spectrometer, operated at the following conditions: collimator - 10 mm; atmosphere - vacuum; tube voltage - 50 kV; tube flow - automatic  $\mu\text{A}$ ; irradiation time - 100 s; acquisition mode - quantitative/FP; analytical line -  $\text{K}\alpha$ .

The mean values of dispersion, stability, and compressive strength of a total of 500 subsamples were determined, as follows: 250 subsamples of plinthite and 250 subsamples of petroplinthite. The comparisons were performed using non-parametric statistics, based on the mean confidence interval ( $\text{CI} = 95 \%$ ;  $p < 0.05$ ) and the mean standard error ( $\pm \text{SE}$ ) was used to test the reliability of the calculated sample mean, using software Xlstat (Addinsoft, 2016). The data were organized in figures and tables with Microsoft Excel 2010.

## RESULTS AND DISCUSSION

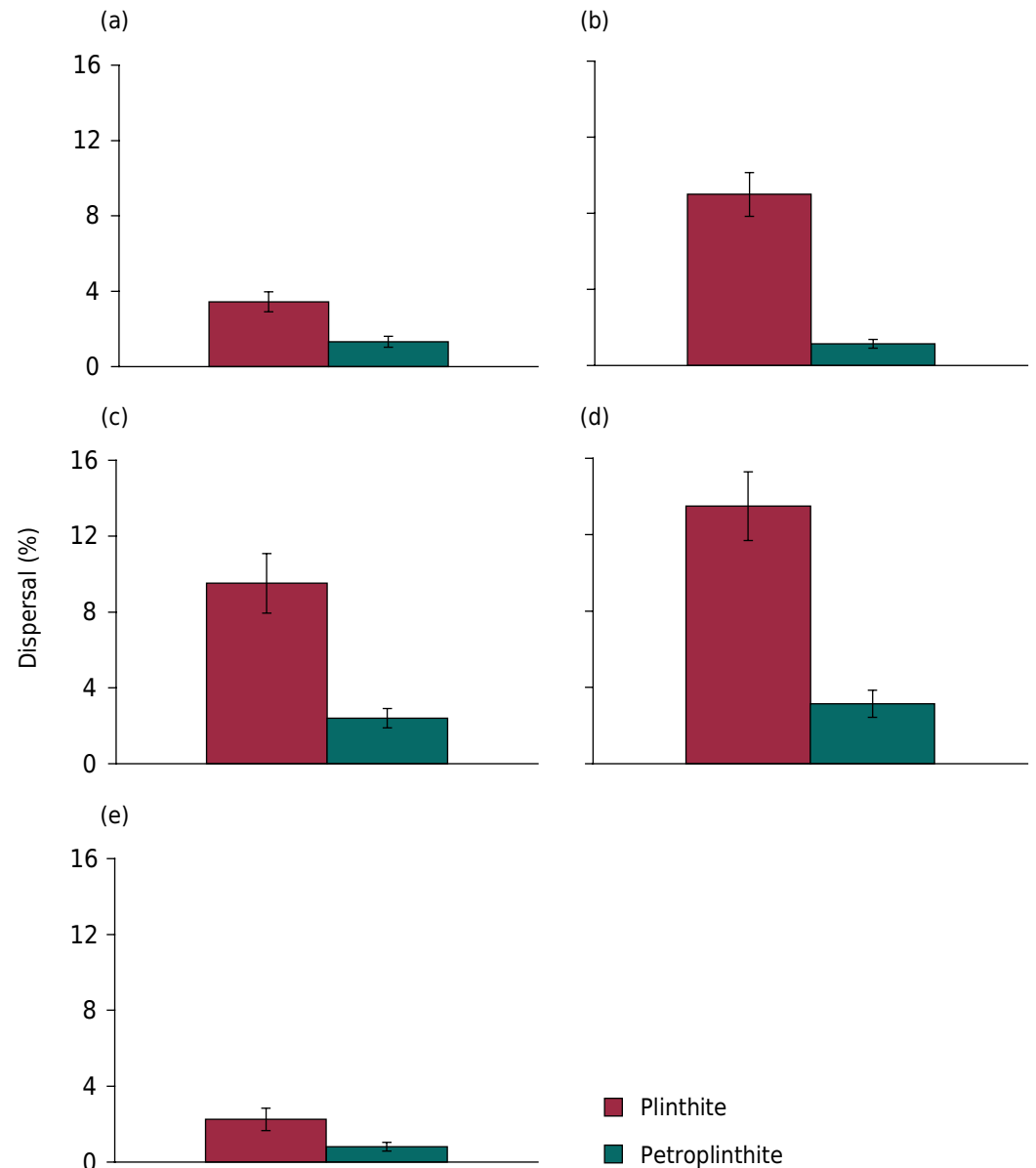
### Degree of dispersion of plinthite and petroplinthite in response to different treatments and immersion periods

Dispersion of ferruginous materials under different immersion treatments is generally affected (Figure 1). Petroplinthite indicated less dispersion, i.e., a smaller amount of material was dispersed than for plinthite, in all treatments. This result is mainly related to the higher content of Fe oxides in petroplinthite (Table 4). Iron oxides are cementing agents and, therefore, do not facilitate the dispersion of ferruginous materials (Donagemma et al., 2003; Rodrigues et al., 2011). Factors such as isomorphic substitution (Fe for Al) and crystallinity degree of Fe oxides possibly contributed to the low degree of dispersion (Santos and Batista, 1996; Ker, 1997).

For both materials, the highest dispersion degree was observed in the subsamples immersed in sodium hydroxide solution + sodium hexametaphosphate, however not differing from petroplinthite in the sodium hydroxide treatment (Figure 2). This result is associated to the action of the reagents, sometimes in increasing the pH of the solution (sodium ions) and sometimes resulting in negative charges (phosphate ions) on the surface of the oxides by the reaction of ions with the hydrogen of the functional groups, with consequent improvement in dispersion (Rodrigues et al., 2011).

Plinthite and petroplinthite are not completely inert and react or are dispersed by hydrolysis, indicating a certain degree of reversibility of the hardening process when subjected to the action of different agents. Medium acidification (use of acid solution) did not induce a high degree of dispersion of plinthite and petroplinthite. In this case, both materials are more stable at these acidity levels, close to those of their formation environment (Table 3). Regarding plinthite, there was also a considerable degree of dispersion after immersion in calcium carbonate and sodium hydroxide solutions. These results indicate that in limed or salinization-prone environments, the dispersion process of plinthite or petroplinthite can be accelerated.

Our studies did not allow the identification of the sections of plinthite and petroplinthite subsamples with highest dispersion degree, since the hardness, composition, and arrangement of these materials in a given soil horizon is variable, due to the great variability



**Figure 1.** Mean value and standard error of the mean plinthite and petroplinthite dispersion of the studied profiles, immersed in distilled water (a), calcium carbonate (b), sodium hydroxide (c), sodium hydroxide + sodium hexametaphosphate (d), and acid solution (e) (95 % confidence interval;  $p < 0.05$ ).

in the subsamples. In spite of the precautions taken with the separation (division) of the materials, the compositions and shapes of plinthite and petroplinthite subsamples were variable. Therefore, it is believed that the dispersion process observed in these materials is primarily related to forms of petroplinthite in more recent development stages or with lower Fe contents, which occur in each sampling unit. This fact also explains the larger amount of dispersed material of plinthite than petroplinthite.

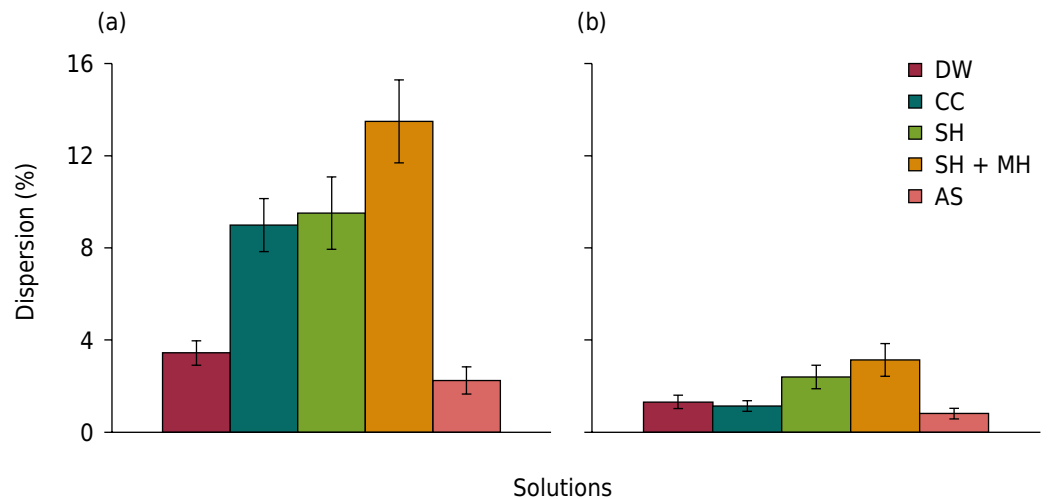
The immersion periods of each treatment had no effect on the dispersion of plinthite and petroplinthite, but the dispersion values between the materials differed (Table 5). The variations observed among the different periods for each treatment are attributed to the differences in the natural composition of each subsample.

In short, the duration of immersion had no effect on the amount of dispersed material in all treatments. Therefore, the time factor has little or no effect on the dispersion of plinthite and petroplinthite in comparison with the reagent factor.

**Table 4.** Main chemical components of plinthite (PL) and petroplinthite (PP) determined by X-ray fluorescence spectrometry (XRF) for each studied profile

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	MgO	K <sub>2</sub> O	V <sub>2</sub> O <sub>5</sub>	ZrO <sub>2</sub>
%								
Profile 2 - <i>Plintossolo Argilúvico Eutrófico petroplíntico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)								
PL	55.53	21.81	21.23	0.98	n.a.	n.a.	0.05	0.08
Profile 5 - <i>Plintossolo Pétrico Concrecionário típico</i> (Santos et al., 2013)/Plinthosol (WRB, 2015)								
PP	42.55	17.74	38.77	0.68	n.a.	n.a.	0.13	0.05

n.a. = not analyzed.


**Figure 2.** Mean value and standard error of the mean dispersion of plinthite (a) and petroplinthite (b) (95 % confidence interval;  $p < 0.05$ ). DW: distilled water; CC: calcium carbonate; SH: sodium hydroxide; SH + HM: sodium hydroxide + sodium hexametaphosphate; AS: acid solution.

**Table 5.** Mean and standard error of the mean dispersion of plinthite and petroplinthite subsamples in the soil profiles, immersed in different solutions for different periods

Material	Period	DW	CC	SH	SH + HM	AS
		Dispersion				
		%				
Plinthite	20	3.01 ± 0.34 <sup>(1)</sup>	10.34 ± 1.35	10.86 ± 1.71	12.17 ± 1.01	2.07 ± 0.78
Plinthite	40	4.36 ± 0.83	8.33 ± 0.85	7.56 ± 1.53	14.47 ± 1.93	2.82 ± 0.42
Plinthite	80	3.06 ± 0.39	9.60 ± 0.74	8.60 ± 1.37	15.47 ± 2.51	1.74 ± 0.50
Plinthite	160	3.35 ± 0.17	7.70 ± 1.37	11.01 ± 1.37	11.85 ± 1.16	2.35 ± 0.60
Petroplinthite	20	1.36 ± 0.24	1.24 ± 0.28	2.24 ± 0.64	4.19 ± 0.98	0.92 ± 0.32
Petroplinthite	40	1.23 ± 0.41	0.95 ± 0.21	2.38 ± 0.36	2.78 ± 0.77	0.73 ± 0.18
Petroplinthite	80	1.29 ± 0.32	0.93 ± 0.18	2.70 ± 0.63	2.84 ± 0.30	0.92 ± 0.24
Petroplinthite	160	1.42 ± 0.19	1.43 ± 0.20	2.29 ± 0.39	2.76 ± 0.55	0.68 ± 0.16

<sup>(1)</sup> 95 % confidence interval ( $p < 0.05$ ). DW: distilled water; CC: calcium carbonate; SH: sodium hydroxide; SH + HM: sodium hydroxide + sodium hexametaphosphate; AS: acid solution.

### Degree of stability of plinthite and petroplinthite in response to different treatments and immersion periods

Regarding the degree of stability of the plinthite and petroplinthite subsamples previously subjected to the dispersion test, both remained practically unchanged when the subsamples were passed through a 2.00 mm mesh sieve, with little distribution when sieved through smaller mesh. This indicates high stability of the ferruginous materials examined here (Table 6). According to Momoli and Cooper (2016), the cementing action of Fe and Al oxides accounts for the high cohesion between particles, favoring stabilization, which confirms our results.

**Table 6.** Mean value and standard error of the mean stability of the plinthite and petroplinthite subsamples in the soil profiles, immersed in different solutions

Material	Set of sieves					
	2.00 mm	1.00 mm	0.50 mm	0.25 mm	0.106 mm	<0.106 mm
Stability <sup>(1)</sup>						
%						
Distilled water						
Plinthite	99.61 ± 0.05 <sup>(2)</sup>	0.01 ± 0.01	0.00 ± 0.00	0.00 ± 0.00	0.13 ± 0.03	0.25 ± 0.04
Petroplinthite	99.71 ± 0.07	0.03 ± 0.02	0.00 ± 0.00	0.04 ± 0.03	0.08 ± 0.03	0.14 ± 0.05
Calcium carbonate						
Plinthite	99.27 ± 0.08	0.05 ± 0.02	0.02 ± 0.01	0.01 ± 0.01	0.47 ± 0.09	0.18 ± 0.06
Petroplinthite	99.36 ± 0.05	0.01 ± 0.01	0.00 ± 0.00	0.05 ± 0.02	0.47 ± 0.05	0.11 ± 0.03
Sodium hydroxide						
Plinthite	99.26 ± 0.07	0.11 ± 0.04	0.01 ± 0.01	0.04 ± 0.01	0.29 ± 0.07	0.29 ± 0.06
Petroplinthite	99.62 ± 0.04	0.12 ± 0.03	0.05 ± 0.02	0.03 ± 0.02	0.08 ± 0.03	0.10 ± 0.03
Sodium hydroxide + Sodium hexametaphosphate						
Plinthite	99.31 ± 0.06	0.09 ± 0.04	0.03 ± 0.02	0.07 ± 0.03	0.30 ± 0.06	0.20 ± 0.06
Petroplinthite	99.44 ± 0.05	0.12 ± 0.05	0.05 ± 0.02	0.04 ± 0.02	0.23 ± 0.04	0.12 ± 0.03
Acid solution						
Plinthite	99.47 ± 0.08	0.05 ± 0.04	0.03 ± 0.03	0.05 ± 0.03	0.20 ± 0.07	0.20 ± 0.06
Petroplinthite	99.58 ± 0.05	0.00 ± 0.00	0.04 ± 0.02	0.07 ± 0.03	0.21 ± 0.05	0.10 ± 0.03

<sup>(1)</sup> The stability of plinthite and petroplinthite subsamples was analyzed through wet sieving on a Yoder shaker apparatus (Donagema et al., 2011).

<sup>(2)</sup> 95 % confidence interval ( $p < 0.05$ ).

Practically no reversibility of the hardening process of the ferruginous materials was observed in response to the action of different chemical agents, suggesting that this process is irreversible for the region and for the tested periods. However, a small fraction of material detached from the plinthite and petroplinthite subsamples was retained when a smaller mesh sieve (<0.106 mm) was used, and this fact cannot be dissociated from the action of chemical dispersants, with a higher or lower impact on the structural stability of the less cemented minerals present in these materials. Given that the plinthite and petroplinthite units were not entirely purified or uniform, i.e., not free from portions of the soil matrix or mottles adhered internally, it is believed that this impact on the structural stability is related to the action of chemical agents on portions with less or less efficient “welding”, an inherent property of this type of material, and thus the most “mature” portions, i.e., the proper plinthite and petroplinthite, remained unchanged.

Regarding the effect of the immersion periods, no behavioral trend could be detected based on the results (Table 7). The small differences observed in the assessed materials can be attributed to the natural variability in composition.

### Compressive strength of plinthite and petroplinthite under different treatments and immersion periods

The compressive strength applied to the plinthite subsamples was much lower than that applied to the petroplinthite subsamples in all treatments of this study (Figure 3). This finding was already expected, due to the lower total Fe contents of plinthite, related to a more recent genesis. According to Garcia et al. (2013), plinthite also has secondary minerals in the hardening process, such as not completely crystalline kaolinite or Fe oxides, which are still susceptible to dispersive or disaggregating action. In ferruginous materials, Santos and Batista (1996) found values that allow concluding that the compressive strength applied to the plinthites of this study reflect a negative association between the most crystalline Fe forms and the hardening degree of these structures. Similarity, Martins et al. (2018) verified a strong correlation between the Fe contents and the compressive strength applied in plinthite samples of soil profiles of the Araguaia river floodplain.

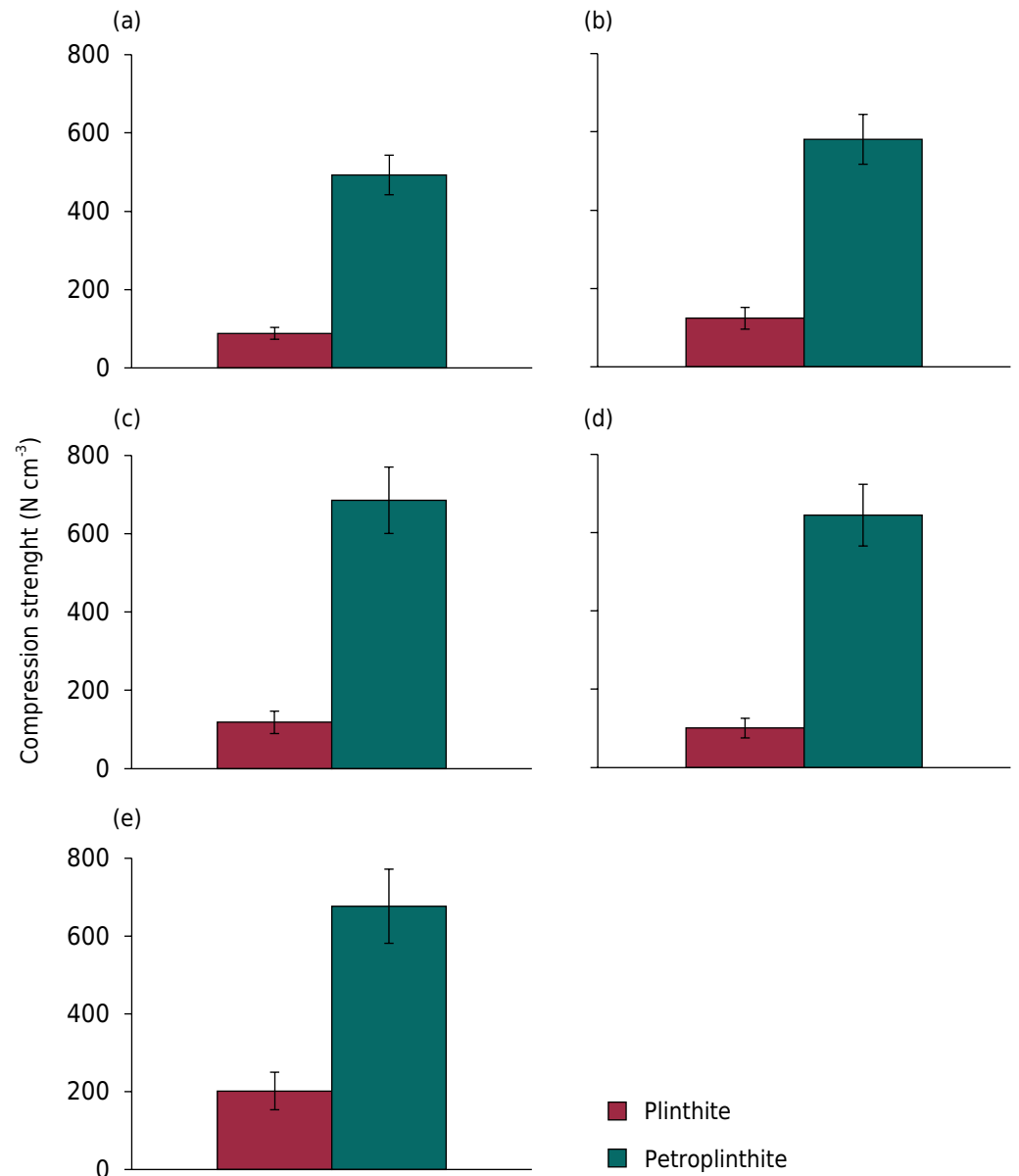


**Table 7.** Mean value and standard error of the mean stability of plinthite and petroplinthite of the profiles, immersed in different solutions for different periods

Material	Period	Set of sieves					
		2.00 mm	1.00 mm	0.50 mm	0.25 mm	0.106 mm	<0.106 mm
		Stability <sup>(1)</sup>					
day		%					
Distilled water							
Plinthite	20	99.58 ± 0.04 <sup>(2)</sup>	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.02 ± 0.02	0.40 ± 0.04
Plinthite	40	99.86 ± 0.05	0.05 ± 0.05	0.00 ± 0.00	0.00 ± 0.00	0.03 ± 0.02	0.06 ± 0.03
Plinthite	80	99.33 ± 0.11	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.34 ± 0.07	0.33 ± 0.09
Plinthite	160	99.67 ± 0.05	0.00 ± 0.00	0.01 ± 0.01	0.00 ± 0.00	0.13 ± 0.03	0.19 ± 0.05
Petroplinthite	20	99.28 ± 0.14	0.05 ± 0.03	0.00 ± 0.00	0.12 ± 0.10	0.20 ± 0.08	0.35 ± 0.13
Petroplinthite	40	100.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
Petroplinthite	80	99.54 ± 0.08	0.08 ± 0.07	0.02 ± 0.02	0.03 ± 0.03	0.12 ± 0.07	0.21 ± 0.08
Petroplinthite	160	100.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
Calcium carbonate							
Plinthite	20	99.63 ± 0.03	0.02 ± 0.02	0.00 ± 0.00	0.02 ± 0.02	0.22 ± 0.06	0.11 ± 0.06
Plinthite	40	99.53 ± 0.08	0.01 ± 0.01	0.00 ± 0.00	0.00 ± 0.00	0.37 ± 0.09	0.09 ± 0.06
Plinthite	80	98.91 ± 0.14	0.03 ± 0.03	0.06 ± 0.05	0.03 ± 0.03	0.96 ± 0.17	0.01 ± 0.01
Plinthite	160	99.01 ± 0.15	0.13 ± 0.09	0.01 ± 0.01	0.01 ± 0.01	0.34 ± 0.23	0.50 ± 0.20
Petroplinthite	20	99.62 ± 0.05	0.02 ± 0.02	0.00 ± 0.00	0.04 ± 0.04	0.28 ± 0.07	0.04 ± 0.04
Petroplinthite	40	99.44 ± 0.08	0.00 ± 0.00	0.00 ± 0.00	0.12 ± 0.05	0.42 ± 0.07	0.02 ± 0.02
Petroplinthite	80	99.15 ± 0.10	0.00 ± 0.00	0.00 ± 0.00	0.04 ± 0.04	0.63 ± 0.12	0.18 ± 0.08
Petroplinthite	160	99.24 ± 0.08	0.02 ± 0.02	0.02 ± 0.02	0.00 ± 0.00	0.55 ± 0.05	0.17 ± 0.07
Sodium hydroxide							
Plinthite	20	99.30 ± 0.09	0.00 ± 0.00	0.00 ± 0.00	0.02 ± 0.02	0.47 ± 0.14	0.21 ± 0.10
Plinthite	40	99.24 ± 0.12	0.06 ± 0.06	0.02 ± 0.02	0.06 ± 0.04	0.30 ± 0.13	0.32 ± 0.08
Plinthite	80	98.95 ± 0.14	0.16 ± 0.11	0.03 ± 0.03	0.03 ± 0.03	0.37 ± 0.19	0.46 ± 0.19
Plinthite	160	99.56 ± 0.09	0.21 ± 0.11	0.00 ± 0.00	0.03 ± 0.03	0.03 ± 0.03	0.17 ± 0.08
Petroplinthite	20	99.65 ± 0.05	0.15 ± 0.07	0.10 ± 0.08	0.00 ± 0.00	0.00 ± 0.00	0.10 ± 0.05
Petroplinthite	40	99.59 ± 0.06	0.10 ± 0.06	0.02 ± 0.02	0.00 ± 0.00	0.14 ± 0.05	0.15 ± 0.08
Petroplinthite	80	99.43 ± 0.04	0.15 ± 0.05	0.04 ± 0.02	0.12 ± 0.06	0.21 ± 0.07	0.05 ± 0.04
Petroplinthite	160	99.81 ± 0.08	0.07 ± 0.05	0.03 ± 0.03	0.01 ± 0.01	0.00 ± 0.00	0.08 ± 0.05
Sodium hydroxide + Sodium hexametaphosphate							
Plinthite	20	99.47 ± 0.01	0.00 ± 0.00	0.00 ± 0.00	0.03 ± 0.03	0.33 ± 0.09	0.17 ± 0.09
Plinthite	40	99.46 ± 0.09	0.00 ± 0.00	0.04 ± 0.04	0.00 ± 0.00	0.29 ± 0.11	0.21 ± 0.12
Plinthite	80	98.95 ± 0.10	0.00 ± 0.00	0.00 ± 0.00	0.12 ± 0.08	0.56 ± 0.13	0.37 ± 0.18
Plinthite	160	99.36 ± 0.11	0.36 ± 0.14	0.09 ± 0.06	0.14 ± 0.07	0.02 ± 0.02	0.03 ± 0.03
Petroplinthite	20	99.42 ± 0.04	0.07 ± 0.03	0.00 ± 0.00	0.00 ± 0.00	0.36 ± 0.10	0.15 ± 0.10
Petroplinthite	40	99.57 ± 0.03	0.02 ± 0.02	0.05 ± 0.04	0.00 ± 0.00	0.21 ± 0.05	0.15 ± 0.06
Petroplinthite	80	99.21 ± 0.17	0.33 ± 0.18	0.05 ± 0.03	0.07 ± 0.07	0.26 ± 0.06	0.08 ± 0.04
Petroplinthite	160	99.57 ± 0.08	0.06 ± 0.04	0.09 ± 0.06	0.10 ± 0.06	0.06 ± 0.06	0.12 ± 0.06
Acid solution							
Plinthite	20	99.58 ± 0.11	0.00 ± 0.00	0.00 ± 0.00	0.10 ± 0.10	0.24 ± 0.12	0.08 ± 0.08
Plinthite	40	98.98 ± 0.10	0.18 ± 0.18	0.10 ± 0.10	0.08 ± 0.08	0.46 ± 0.18	0.20 ± 0.13
Plinthite	80	99.30 ± 0.08	0.01 ± 0.01	0.03 ± 0.03	0.02 ± 0.02	0.10 ± 0.10	0.54 ± 0.13
Plinthite	160	100.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
Petroplinthite	20	99.67 ± 0.05	0.00 ± 0.00	0.02 ± 0.02	0.13 ± 0.08	0.07 ± 0.04	0.11 ± 0.07
Petroplinthite	40	99.66 ± 0.09	0.00 ± 0.00	0.06 ± 0.06	0.00 ± 0.00	0.17 ± 0.08	0.11 ± 0.06
Petroplinthite	80	99.65 ± 0.09	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.25 ± 0.10	0.10 ± 0.05
Petroplinthite	160	99.36 ± 0.14	0.00 ± 0.00	0.07 ± 0.07	0.14 ± 0.08	0.36 ± 0.15	0.07 ± 0.05

<sup>(1)</sup> The stability of plinthite and petroplinthite subsamples was analyzed through wet sieving on a Yoder shaker apparatus (Donagema et al., 2011).

<sup>(2)</sup> 95 % confidence interval (p<0.05).



**Figure 3.** Mean value and standard error of the mean compressive strength of plinthite and petroplinthite of the studied profiles, immersed in distilled water (a), calcium carbonate (b), sodium hydroxide (c), sodium hydroxide + sodium hexametaphosphate (d), and acid solution (e) (95 % confidence interval;  $p < 0.05$ ).

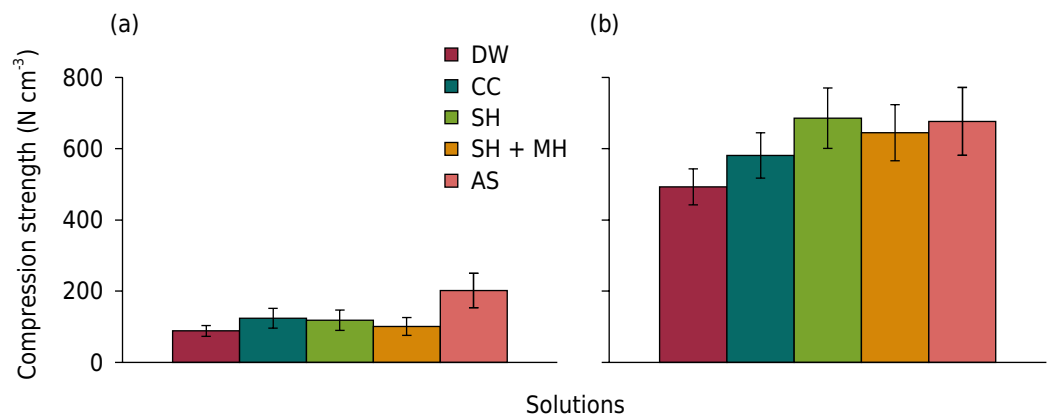
In the case of plinthite, the lowest compressive strength was applied to the subsamples immersed in distilled water and in solutions of calcium carbonate, sodium hydroxide, and sodium hydroxide + sodium hexametaphosphate, without differences between them (Figure 4a). The highest compressive strength (greatest hardness) was observed in the subsamples immersed in acid solution. Thus, it can be seen that plinthite stabilizes in acid solution, or rather, remains stabilized, because the acidity of the medium is similar to the natural conditions of the site of formation, and consequently will not favor its softening (Table 3). On the other hand, the other wetting treatments, all with a pH ranging from neutral to alkaline, indicated a higher softening response, although not directly proportional to the alkalinity of the medium.

For petroplinthite, the lowest compressive strength was observed in the subsamples immersed in distilled water, however not differing from the calcium carbonate solution (Figure 4b). This result may at first indicate that unlike plinthite, petroplinthite is slightly more vulnerable to the action of distilled water (reduced environment) or to low-alkalinity solutions, close to neutrality, and unsusceptible to acid or highly alkaline solutions. Similar

results were observed by Alexander and Cady (1962) in Sierra Leone, Africa, where it was found that the softest laterite was found near creeks, due to the higher availability of moisture and vegetation cover.

Considering data of the strength applied to petroplinthite subsamples in the different treatments (Table 8), however, the differences can be explained by differences in the composition of the subsamples rather than by chemical reactions to the treatments, due to the heterogeneity of strengths within a treatment and among treatments as well as the proximity of the values. Moreover, the studied petroplinthite material is completely stabilized and less vulnerable to the action of dispersing agents because its formation occurred in the past. Petroplinthite also has a longer and more uniform maturation stage, which results in higher content and crystallinity of Fe and Al (Miguel et al., 2013).

In general, there was no effect of the duration of immersion on the decrease in the compressive strength applied to plinthite and petroplinthite in the treatments. The great variability in compressive strength between immersion periods is related to the great natural variability observed in the composition of subsamples from a same soil horizon and to the impossibility of selecting subsamples with the same degree of purity because of their different stages of development, consistent with observations by Santos and Batista (1996) and Martins et al. (2018). However, Alexander and Cady (1962) and



**Figure 4.** Mean value and standard error of the mean compressive strength of plinthite (a) and petroplinthite (b) with a 95 % confidence interval ( $p < 0.05$ ). DW: distilled water; CC: calcium carbonate; SH: sodium hydroxide; SH + HM: sodium hydroxide + sodium hexametaphosphate; AS: acid solution.

**Table 8.** Mean value and standard error of the mean compressive strength of the plinthite and petroplinthite subsamples in the soil profiles, immersed in different solutions for different periods

Material	Period	DW	CC	SH	SH + HM	AS
		Strength				
		N cm <sup>-3</sup>				
Plinthite	20	94.56 ± 11.35 <sup>(1)</sup>	76.23 ± 10.78	101.17 ± 16.88	76.01 ± 15.74	133.01 ± 23.11
Plinthite	40	75.09 ± 12.42	97.69 ± 12.39	154.41 ± 45.75	147.24 ± 28.58	225.62 ± 45.99
Plinthite	80	121.31 ± 14.09	146.12 ± 31.24	106.35 ± 24.44	54.26 ± 10.62	114.30 ± 29.86
Plinthite	160	62.32 ± 12.99	175.82 ± 32.16	111.75 ± 14.76	126.11 ± 23.96	334.42 ± 36.55
Petroplinthite	20	483.47 ± 51.22	593.72 ± 41.94	707.61 ± 67.82	497.15 ± 50.61	702.39 ± 131.05
Petroplinthite	40	472.29 ± 67.23	525.69 ± 66.39	557.36 ± 46.33	793.92 ± 63.78	573.45 ± 75.06
Petroplinthite	80	536.95 ± 49.93	518.67 ± 37.14	595.54 ± 74.87	539.20 ± 55.18	568.02 ± 19.72
Petroplinthite	160	478.42 ± 28.93	685.94 ± 82.21	881.93 ± 88.42	749.02 ± 76.53	862.82 ± 77.17

<sup>(1)</sup> 95 % confidence interval ( $p < 0.05$ ). DW: distilled water; CC: calcium carbonate; SH: sodium hydroxide; SH + HM: sodium hydroxide + sodium hexametaphosphate; AS: acid solution.

Daugherty and Arnould (1982) claimed that the conditions required for the hardening of plinthic materials are extremely variable, particularly the amount of time required, in disagreement with the results of this study.

## CONCLUSIONS

Hardness of plinthite and petroplinthite, particularly the former, is susceptible to the action of different chemical agents, especially with alkaline character.

Immersion of plinthite samples in calcium carbonate and sodium hydroxide solutions induced considerable dispersion, suggesting that in limed or salinization-prone environments, the dispersion process of plinthite or petroplinthite can be accelerated.

Structural stability of the plinthite and petroplinthite subsamples, exposed to the action of different chemical agents, was not degraded in the wet aggregate stability test, indicating no reversibility in the hardening process of the materials of the Araguaia River floodplain, under the conditions of this study.

Immersion periods in all treatments did not affect the compressive strength applied to the plinthite and petroplinthite samples.

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## REFERENCES

- Addinsoft. Xlstat version 2016.4. Software e guia do usuário. New York: 2016.
- Alexander LT, Cady JG. Genesis and hardening of laterite in soils. Washington, DC: United States Department of Agriculture; 1962. (Technical bulletin, 1282).
- Brasil. Ministério das Minas e Energia. Secretaria Geral. Projeto Radambrasil: Folha SD. 22 Goiás: geologia, geomorfologia, pedologia, vegetação e uso potencial da terra. Rio de Janeiro: 1981. (Levantamento dos recursos naturais, 25).
- Cardoso MRD, Marcuzzo FFN, Barros JR. Classificação climática de Köppen-Geiger para o estado de Goiás e o Distrito Federal. *Acta Geogr.* 2014;8:40-55. <https://doi.org/10.5654/actageo2014.0004.0016>
- Coelho MR, Vidal-Torrado P, Ladeira FSB. Macro e micromorfologia de ferricretes nodulares desenvolvidos de arenito do Grupo Bauru, Formação Adamantina. *Rev Bras Cienc Solo.* 2001;25:371-85. <https://doi.org/10.1590/S0100-06832001000200013>
- Daniels RB, Perkins HF, Hajek BF, Gamble EE. Morphology of discontinuous phase plinthite and criteria for its field identification in the southeastern United States. *Soil Sci Soc Am J.* 1978;42:944-9. <https://doi.org/10.2136/sssaj1978.03615995004200060024x>
- Daugherty LA, Arnold RW. Mineralogy and iron characterization of plinthitic soils on alluvial land-forms in Venezuela. *Soil Sci Soc Am J.* 1982;46:1244-52. <https://doi.org/10.2136/sssaj1982.03615995004600060025x>
- Donagema GK, Campos DVB, Calderano SB, Teixeira WG, Viana JHM. Manual de métodos de análise do solo. 2. ed. rev. Rio de Janeiro: Embrapa Solos; 2011.
- Donagemma GK, Ruiz HA, Fontes MPF, Ker JC, Schaeffer CEGR. Dispersão de Latossolos em resposta à utilização de pré-tratamentos na análise textural. *Rev Bras Cienc Solo.* 2003;27:765-72. <https://doi.org/10.1590/S0100-06832003000400021>
- Eze PN, Udeigwe TK, Meadows ME. Plinthite and its associated evolutionary forms in soils and landscapes: a review. *Pedosphere.* 2014;24:153-66. [https://doi.org/10.1016/S1002-0160\(14\)60002-3](https://doi.org/10.1016/S1002-0160(14)60002-3)

Garcia CHP, Lima HN, Silva FWR, Neves Junior AF, Teixeira WG, Macedo RS, Tavares SG. Chemical properties and mineralogy of soils with plinthite and petroplinthite in Iranduba (AM), Brazil. *Rev Bras Cienc Solo*. 2013;37:936-46. <https://doi.org/10.1590/S0100-06832013000400011>

Gardi C, Angelini M, Barceló S, Comerma J, Gaistardo CC, Rojas AE, Jones A, Krasilnikov P, Brefin MLMS, Montanarella L, Ugarte OM, Schad P, Rodríguez MIV, Vargas R. Atlas de suelos de América Latina y el Caribe. Luxembourg: Comisión Europea - Oficina de Publicaciones de la Unión Europea; 2014.

Instituto Brasileiro de Geografia e Estatística - IBGE. Manual técnico de pedologia. 3. ed. Rio de Janeiro, RJ: IBGE; 2015. (Manuais técnicos em geociências).

IUSS Working Group WRB. World reference base for soil resources 2014, update 2015: International soil classification system for naming soils and creating legends for soil maps. Rome: Food and Agriculture Organization of the United Nations; 2015. (World Soil Resources Reports, 106).

Ker JC. Latossolos do Brasil: uma revisão. *Geonomos*. 1997;5:17-40. <https://doi.org/10.18285/geonomos.v5i1.187>

Martins APB, Santos GG, Oliveira VA, Maranhão DDC, Collier LS. Hardening and stability of plinthic materials of the Araguaia river floodplain under different drying treatments. *Rev Bras Cienc Solo*. 2018;42:e0170190. <https://doi.org/10.1590/18069657rbcs20170190>

Miguel P, Dalmolin RSD, Pedron FA, Fink JR, Moura-Bueno JM. Caracterização de plintitas e petroplintitas em solos da Depressão Central do Rio Grande do Sul. *Cienc Rural*. 2013;43:999-1005. <https://doi.org/10.1590/S0103-84782013005000065>

Momoli RS, Cooper M. Erosão hídrica em solos cultivados e sob mata ciliar. *Pesq Agropec Bras*. 2016;51:1295-305. <https://doi.org/10.1590/s0100-204x2016000900029>

Ogunwole JO, Esu IE, Kparmwang T, Chude VO. Effects of prolonged shaking periods and contents of iron oxides on dispersion of plinthitic soil in Nigeria. *Commun Soil Sci Plan*. 2001;32:2293-306. <https://doi.org/10.1081/CSS-120000284>

Rodrigues C, Oliveira VA, Silveira PM, Santos GG. Chemical dispersants and pre-treatments to determine clay in soils with different mineralogy. *Rev Bras Cienc Solo*. 2011;35:1589-96. <https://doi.org/10.1590/S0100-06832011000500013>

Santos HG, Jacomine PKT, Anjos LHC, Oliveira VA, Oliveira JB, Coelho MR, Lumberras JF, Cunha TJF. Sistema brasileiro de classificação de solos. 3. ed. rev. ampl. Rio de Janeiro: Embrapa Solos; 2013.

Santos MC, Batista MA. Avaliações física, química e mineralógica em solos plínticos da região meio-norte do Brasil, submetidos a teste de umedecimento e secagem. *Rev Bras Cienc Solo*. 1996;20:21-31.

Santos RD, Santos HG, Ker JC, Anjos LHC, Shimizu SH. Manual de descrição e coleta de solo no campo. 7. ed rev ampl. Viçosa, MG: Sociedade Brasileira de Ciência do Solo; 2015.

Soil Survey Staff. Keys to soil taxonomy. 12th ed. Washington, DC: United States Department of Agriculture, Natural Resources Conservation Service; 2014.

Zhang M, Karathanasis AD. Characterization of iron-manganese concretions in Kentucky Alfisols with perched water tables. *Clay Clay Miner*. 1997;45:428-39. <https://doi.org/10.1346/CCMN.1997.0450312>