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Comparison of methods for the evaluation of the fertility of glycols in northern Brazil

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Abstract

The predominant soils of the Amazon estuary are the haplic and salic gleisols, found in large areas. The objective was to compare analyzes of wet soil with those of pre-dried samples, and for wet samples they were adjusted using a conversion factor. Soil samples from two different areas of the Amazon estuary in northern Brazil. The soil samples were immersed in distilled water (four replicates and two soils) for a period of 132 days, with observations for a period of 52 days. Chemical analysis of P, Fe, Mn, Ca, K, Zn and Cu - was analyzed using both methods (wet and pre-dry). The results of both procedures were able to detect the variation in the concentrations of the different elements over the time of immersion. The increased sensitivity of the wet soil approach in these specific cases represents a major advantage in determining the chemical characteristics of the soils found in the analyzed environments.

Keywords: Amazon, soil, estuary, chemical characteristics

1. Introduction

In Brazil, the chemical analysis of soils began in the 1960s, accompanying the expansion of industrialized agriculture in the country (Vetori, 1969; Guimarães *et al.*, 1970; Bloise and Moreira, 1976; Embrapa, 1979) [41, 18, 4, 12]. Initially, analytical approaches were adapted from those used internationally (Jackson, 1958; Chapman and Pratt, 1961; Black, 1965) [20, 10, 3]. These procedures were eventually standardized by the Brazilian Agricultural Research Enterprise, EMBRAPA (EMBRAPA, 1979, 1997) [12, 11]. In 1975, the Brazilian Society for Soil Sciences (SNCS) recommended the application of units of the international system (SI), with the aim of standardizing the terminology used for the analysis of chemical composition and fertility.

Scientists have long recognized the need to standardize procedures in order to guarantee the systematic interpretation of results. This preoccupation begins with the preparation of the samples, and includes the drying of the soil, which must be carried out in a stove or the air, which is then sieved through a 2 mm mesh. The material of less than 2 mm in size is referred to as the active fraction of the soil, and is sent to one of the existing soil laboratories for analysis (Camargo *et al.*, 1986; Embrapa, 1997; Cantarella, 1995) [7, 13, 8]. Such simple procedures facilitate the interpretation of the results, and have been adopted universally by the laboratories, with minor modifications in some cases, which should be informed together with the results.

However, some soils suffer alterations which affect their fertility when they are in contact with water, which changes once again when they dry out. Specifically, the presence of water fills the pores, and excludes the air. A few hours after flooding, the aerobic micro-organisms contained in the soil use all the oxygen dissolved in the water, creating an oxygen-free environment, which is then occupied by facultatively aerobic or strictly anaerobic micro-organisms, which multiply vary rapidly and decompose the organic matter in the soil using the oxidized compounds in the soil to capture electrons, converting the substrate to a reductive condition (Ponnamperuma, 1972; Camargo *et al.*, 1999, Inglett *et al.*, 2012) [28, 6, 19].

This process is characterized by a number of different alterations (Ponnamperuma, 1972, Sanches, 1981; Camargo *et al.*, 1999; Tan, 2011) [28, 29, 6, 40], which include the liberation of Fe and Mn following the reduction of the minerals that contain these elements. This affects indirectly the availability of phosphorus and other elements such as calcium, magnesium, potassium, and sodium, which, while not being reduced, may be displaced to the solution

Through the production of soluble ions. The pH is also altered by the reduction reaction processed in the iron and manganese oxy-hydroxides, which produces hydroxyls, which make the medium less acidic (Catling, 1992, Lima *et al.*, 2005) ^[9, 22]. After drying, however, the oxygen returns to the soil and oxidizes the reduced substances, provoking a reversion of the chemical and electrochemical transformations caused by the flooding (Ponnamperuma, 1972; Fageria, 1984; Assis *et al.*, 2000; Lima *et al.*, 2005) ^[28, 14, 1, 22].

A number of studies have analyzed the soil solution obtained from experimental samples for the evaluation of the effects of water on the soil pores. In this case, the solution is removed from the samples by suction or gravity, and the analysis is conducted without the application of a chemical extractor, although weak acids may be used in some cases (Silva *et al.*, 1988; Melo *et al.*, 1992; Silva *et al.*, 2003; Silva and Ranno, 2005, Lima *et al.*, 2005) ^[38, 25, 34, 33, 22]. The lack of published tables for the interpretation of the adjusted results of this approach and the absence of standards for the dilution of the experimental samples, which must be irrigated in order to maintain the water level, hampers the regular use of this procedure for the analysis of soils affected by inundation.

The measurement of the available elements through the evaluation of the adsorbed portion in the soil, has a number of advantages. The analysis of the exchangeable cations and the non-exchangeable acidity (H^+) provides an estimate of the capacity of the soil for the exchange of cations according to the quantity of these charges, which corresponds to the total of negative charges in the soil. It is possible to assess the saturation of alkalis and aluminum in the soil in a similar fashion. These data, together with the availability of a large number of tables for the interpretation of soil analyses, established for agricultural purposes, facilitate the interpretation of the results of the analysis of these soils (Souza and Lobato, 2004; Jones Jr., 1984; Oleynik, 1984; Lopes and Guilherme, 2004) ^[40, 21, 26, 23].

Given these considerations, a number of authors (Silva, 1995; Silva *et al.*, 1996; Ferreira *et al.*, 1998; Ferreira and Botelho, 1999; Mattar *et al.*, 2002; Silva, 2008) ^[32, 39, 16, 17, 35] have used the humid soil approach, that is, without drying the samples prior to analysis, in order to maintain the characteristics of the samples observed at the moment they were collected. As the results and the interpretation tables are designed for the volume or weight of dry samples, these authors have used a humidity factor to convert the results into values equivalent to those of dry samples. Given this, each humid sample is collected in duplicate, and while one sample is reserved for chemical

analysis, the other is dried in a stove and then dehydrated in a dessicator prior to being weighed. The ratio of dry to humid weight provides a correction factor for the adjustment of the results. However, while this approach has been used in a number of different studies, it is necessary to standardize the procedure by comparing the results of this method systematically with those of the traditional approach, based on the analysis of dry samples. This will amplify perspectives for the collection and routine analysis of hydromorphic soils.

The humid-soil approach developed by Silva, (1995) ^[32], Silva *et al.* (1996) ^[39], Ferreira *et al.* (1998) ^[16], Ferreira and Botelho (1999) ^[17], Mattar *et al.* (2002) ^[24], and Silva (2008) ^[35] is especially relevant to the environments of the northern coast of Brazil, where hydromorphic soils dominated by gleysols are common. Haplic gleysols predominate along the whole of the Brazilian state of Amapá, the estuary of the Amazon River, and the coast of Pará state as far east as the mouth of the Pará River. Salic gleysols predominate along the coastline between this river and the mouth of the Gurupi River on the border of Maranhão state, to the east (Lima *et al.*, 2001) ^[22]. This belt of salic gleysol, known as the macrotidal mangrove coast, underlies the World's largest continuous tract of mangrove forest (Souza, 2004) ^[40]. Haplic gleysols can be found in the rivers that flow into this mangrove system. The constant saturation of these soils caused by the tidal cycle provokes alterations in their fertility, which are generally beneficial (Catling, 1992) ^[9].

The objective of the present study was to compare the results of the analysis (macro- and micronutrients) of humid samples of soils from the várzeas of the Amazon estuary, which were immersed over a 52-day period, with those of pre-dried samples. Subsequently, these procedures were tested for the comparison of the samples of haplic and salic gleysols.

2. Materials and Methods

The study was based on two simultaneous experiments conducted in the Chemical Analyses Laboratory of the Geosciences Institute of the Federal University of Pará (UFPA), which analyzed samples of the gleysols collected from the várzea swamps of the Guamá River in the city of Belém and the mangrove forests of the estuary of the Caeté River, in the vicinity of the town of Bragança, both located in the Brazilian state of Pará (Figure 1). The samples from the Guamá were collected 10 km from the mouth of the river, 500 m from the right bank of the river, while those from the Caeté were collected in the mangrove neighboring Bragança, 5.2 km from the mouth of the river

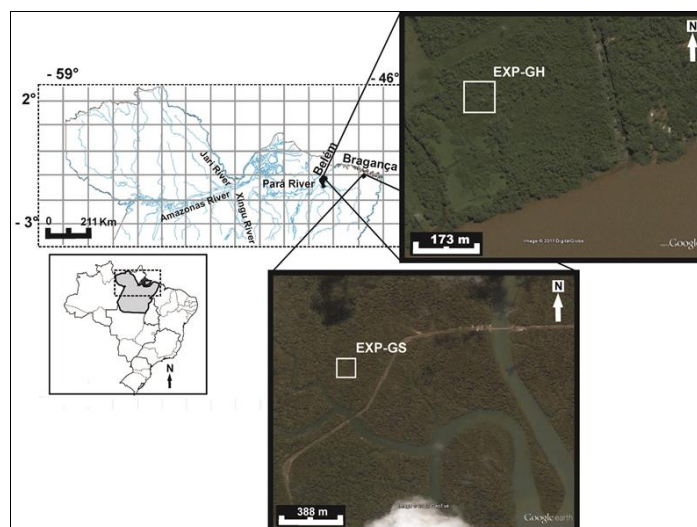


Fig 1: Location of the study sites in the várzea of the Guamá River ($1^{\circ}27'32.50''$ S, $48^{\circ}25'33.89''$ W), where the samples of haplic gleysol were obtained, and the mangrove of the Caeté Estuary ($0^{\circ}50'38.38''$ S, $46^{\circ}38'51.67''$ W), where the samples of salic gleysol were collected.

The samples were collected up to a maximum depth of 20 cm, corresponding to the A1 horizon. To guarantee a representative sample, 65 separate subsamples of approximately 1.5 kg each were collected in Bragança and 63 in Guamá, using a hand drill. The subsamples were homogenized to produce a single composite sample for each site, called EXP-GH (haplic gleysol) and EXP-GS (gleysol salic do Caeté).

The belém floodplains, located on the right bank of the Guamá river, are typical of this type of forest, with plant species such as acapurana (*Campsiandra laurifolia* Benth.), Ubim (*Geonoma baculifera* K.), mamorana (*Pachira aquatica* Aubl.), Ananim (*Symphonia globulifera* L.), straw (*Clitoria arbores* Benth.) And ucuúba (*Virola sebifera* Aubl.). The Caeté mangrove forest is characterized by the presence of red (*Rhizophora mangle* L.), white (*Laguncularia racemosa* L.) and black (*Avicennia* sp L.) mangroves. Soil fertility was determined by standard methods, following Embrapa (1997)^[11]; Chapmam and Pratt (1961)^[10] for both the Guamá river haplic gleysol and Bragança sage gleysol (Silva and Costa, 2012)^[36]. Both types of soil belong to the same pedological category, which is characterized by the

presence of water in the soil profile. The principal difference between these soils is the exposure of the salic gleysol to seawater, which determines the saline characteristics of this substrate, to which the overlying vegetation must be adapted. The mineralogical identification of these soils by X-ray diffraction indicated the presence of quartz, illite, kaolinite, smectite, goethite and anatase in the haplic gleysol, and quartz, kaolinite, illite, smectite, and anatase in the salic gleysol (Silva and Costa, 2011)^[37]. However, while these soils are very similar in mineralogical terms, they present major differences in fertility (Table 1). Surrounded by weathered and leached soils, the gleysols are relatively fertile by local standards (Vieira and Santos, 1987)^[42]. The haplic gleysol is less fertile than the salic gleysol, with base saturation close to 50%, although the salic gleysol is characterized by a saturation of 88.64%, reflecting its eutrophic status.

The presence in the soil of 2:1 clay minerals and organic matter contributes to the high fertility of the substrate, in contrast with the soils of the surrounding area, which have no 2:1 clay minerals and only low concentrations of organic matter.

Table 1: The pH, concentrations of elements and available compounds, and granulometry of the samples EXP-GH and EXP-GS, for the analysis of the fertility of the respective soils (adapted from Silva and Costa, 2011^[37], Silva and Costa, 2012^[36]).

Sample	pH		C		M.O	P	Ca	Mg	K	SB	H	Al	T	V	m	
	H ₂ O	KCl	-----g/kg-----		mg/dm ³	-----cmol/ dm ³ -----										-----%-----
EXP-GH	4.69	3.59	3.9	6.72	2.52	1.52	2.48	0.08	4.08	3.94	1.60	9.62	42.41	28.67		
EXP-GS	6.80	5.55	10.52	18.14	20.76	3.81	12.85	2.10	18.74	2.35	0.05	21.14	88.64	0.26		
Sample	Coarse sand				Fine sand				Silt				Clay			
	-----g/kg-----															
EXP-GH	2.90				28.00				743.00				226.3			
EXP-GS	105.80				528.30				76.60				298.30			

The experimental procedure consisted of placing four samples of each type of soil in 7-liter styrofoam boxes, which were then immersed in distilled water and maintained underwater for 132 days, when the procedure was halted. The study focuses on the first 52 days only. A modified syringe was used to obtain duplicate 10 ml samples of the soil, which were immediately weighed. One of the duplicate samples was used for a soil analysis (Embrapa, 1997)^[11] while the other was dried in a stove, dehydrated in a dessicator, and then weighed. The ratio between the weights of the dry and humid samples was used as the correction factor for the evaluation of the results of the analysis of the humid soil samples. The dry sample was then fractionated for chemical analysis, following Embrapa (1997)^[11]. Aliquots were obtained daily for the first five days, in order to accompany the rapid changes that occur during this initial period, then every second day during the second week, and at weekly intervals during the rest of the experimental period. The chemical analysis determined the concentrations of available and exchangeable phosphorus, potassium, and calcium, as well as the micronutrients iron, manganese, zinc, and copper (Embrapa, 1997)^[11].

The variation in the concentrations of the different components of the soil samples were represented graphically for the 52-day study period. The results were evaluated statistically using the Analysis of Variance (ANOVA), for the two treatments, T₁ – analysis of the humid soil, and T₂ – analysis of the dry soil, run in the SISVAR application (Ferreira, 2007)^[15]. The results of

these analyses were compared using the Tukey test.

3. Results and Discussion

The concentration of organic matter in the soil, which was medium in the salic gleysol and low in the haplic gleysol, increased markedly with the depth of the profile (Silva and Costa, 2011, 2012)^[37, 36], partly because of the slower oxidation of this material in comparison with the aerobic environment, given that the oxygen functions as an electron acceptor (Scholz, 2011)^[30], partly because of the intense biological dynamic of these environments, as well as the absence of electron acceptors for the development of the reductive process. These soils also presented relatively high levels of available phosphorus and reduced saturation of aluminum (Lopes and Guilherme, 2004)^[23]. The two types of soil also present distinct granulometric characteristics. The haplic gleysol can be characterized as a silty loam according to the classification system of Vieira and Vieira (1983)^[43], with 743 g/kg of silt in the samples, whereas the salic gleysol was a clayey-sandy loam, with 634.1 g/kg of sand. These differences in texture are reflected in distinct patterns of water retention and penetration of the soil profiles.

The immersion of the samples resulted in considerable modifications of the fertility of the soil, as indicated by the variation in the concentrations of available and exchangeable elements recorded over the 52 days of the study in both the humid and the dry samples (Tables 2 and 3).

Table 2: Variation in the mean concentrations of available P, Ca, K, Zn, Cu, and Fe in the samples of haplic gleysol (horizon A₁) over the 52 days of immersion under distilled water.

Days	K*	K**	Ca*	Ca**	P*	P**	Fe*	Fe**	Mn*	Mn**	Cu*	Cu**	Zn*	Zn**
	-----cmol/ dm ³ -----				-----mg/dm ³ -----									
1	1.28	0.19	225.25	276.50	7.77	55.60	1048	1162	151.50	293.75	1.25	2	6.75	12
2	1.11	0.18	395.25	404.75	8.91	54.48	1179.25	1844	227	298.50	3.75	3.25	11.25	12.50
3	0.71	0.15	346	356.75	10.58	61.17	1479.75	1343	226.75	243	1	1	12	11.25
4	0.13	0.17	249.25	352.75	13.15	51.63	1787.25	2286.25	309.25	338.25	1	2	11.25	13.25
5	0.37	0.18	243.75	325.37	11.10	73.55	2799.50	1753.25	340.75	322.75	2	2	13.75	11.25
8	1.39	0.18	270.25	298.87	20.63	84.58	2628	2475.50	373.75	349.75	2	2.25	13.25	12.50
10	1.06	0.19	285	351.25	15.42	63.03	2886.25	2586.75	367	359.25	2	2.50	16	15
12	0.17	0.18	496.75	472.87	16.46	82.60	5330	1991.50	324.75	343	2	2	14.50	10.25
15	0.18	0.14	456.50	455.50	23.12	83.84	5806.25	3595	360	354.50	1.25	2	14.50	11.75
17	0.48	0.16	404	409.50	20.96	76.65	6203.25	4522.50	316.25	347	1.75	2	15.25	14.25
19	1.01	0.16	446.25	430.12	18.06	97.59	6952	3857.75	355.75	356.75	2.25	3	18	11.50
23	1.24	0.14	434.75	417.50	38.62	91.27	7165	2556.25	334.50	317.50	2.25	2.25	14.75	10
25	1.04	0.17	426	380	48.66	156.40	6690.25	7644.25	346	321.50	0.75	2	14	21
31	0.15	0.18	390	362.62	119.15	139.45	2539	2221	365	351.50	3.75	6.25	10	6.75
38	0.15	0.17	338.50	364.75	72.32	124.95	3887.75	3051.25	343.75	394.25	1.75	4.25	15.25	11.25
45	0.16	0.16	352.50	383	75.30	22.75	8136.25	8147.25	331.75	368.25	1.25	2.77	24.25	16.50
52	0.16	0.13	353.50	388.75	11.47	40.37	7919.50	7333	526	304	5	2	20	27.25

*humid: result obtained for the humid sample; **dry: result obtained for the dry sample.

Table 3: Variation in the mean concentrations of available P, Ca, K, Zn, Cu, and Fe in the samples of salic gleysol (horizon A₁) over the 52 days of immersion under distilled water

Days	K*	K**	Ca*	Ca**	P*	P**	Fe*	Fe**	Mn*	Mn**	Cu*	Cu**	Zn*	Zn**
	-----cmol/ dm ³ -----				-----mg/dm ³ -----									
1	0.15	1.15	353	432.75	103.18	124.48	1168.25	1590.50	14	9.25	1	1	3	3.75
2	0.15	1.13	369.25	389.75	105.28	144.18	1284	1513.75	6	6.5	1	1.25	4.25	4
3	0.67	0.88	342.25	364.50	111.49	122.62	1387.75	1288.75	7.75	6.75	1	1	3.50	3
4	1.31	1.12	348.50	373.5	110.62	140.50	1311.50	1934	7	8.75	1	1	3	3.50
5	1.31	0.98	279	300.75	142.40	212.98	1720.25	1118.80	8	7	1	1	2.5	1.75
8	0.67	1	291.25	317.75	136.31	74.43	1408.75	1833	6.75	6.25	1	1	3	4
10	0.15	1.03	289.75	338	148.36	36.89	1850	1920	8	1.50	1	1	4.25	4
12	0.39	0.87	456.75	411	173.43	34.44	2084.50	1683	9.25	8.25	1	2.75	3.25	3
15	1.29	1	476.25	447.50	33.04	33.79	1606.25	1493	6.75	7.50	1	1	3.25	3.75
17	1.92	1.21	442	447.50	35.89	39.61	2428.75	2474.25	5.25	6	1	1	3.75	4.50
19	2.62	1.05	461.50	441.50	45.63	32.67	2386	1892	8.50	7.75	4	1	5.25	3.50
23	1.51	1.30	389	390.75	50.77	141.82	2410	1763.50	7.50	7.50	4	1.25	2.25	1
25	1.22	1.09	398	455	62.41	75.92	2134.25	3565	6.50	7.50	1	2.25	2.75	4.50
31	1.29	1.15	400	471	144.05	121.87	1549	1438.77	9	8.25	1	1.5	4.25	5
38	1.43	1.36	373	437.25	87.2	151	2133.75	1560.25	10.75	12	1	1	4	4
45	1.28	1.20	278	440.75	66.12	37.50	4450.50	3130.50	4.75	5.75	1	1	6.75	6.50
52	1.30	1.43	388	421	54.35	62.05	4644	3514.25	5	5	1	1	5	5

*humid: result obtained for the humid sample; **dry: result obtained for the dry sample.

The results of the ANOVA indicate significant variations in the concentrations of all the elements in both experiments (Tables 4 and 5). Significant differences were also found between the humid and dry samples for the majority of the elements

analyzed. However, no significant variation was found for calcium, in the haplic gleysol, or for phosphorus, potassium, zinc or manganese, in the salic gleysol, reflecting specific differences in the two types of soil.

Table 4: Results of the ANOVA for the effects of immersion with distilled water on the haplic gleysol samples over time (days) and the two treatments.

VF	DF	Ca	K	P	Zn	Mn	Cu	Fe
		-----cmol/ dm ³ -----		-----mg/dm ³ -----				
Day	16	9.644**	7.61**	12.598**	20.73**	16.086**	8.177**	125.335**
Treatment	1	2.384 ^{NS}	111.188**	126.096**	5.711*	8.899*	12.501**	78.259**
Treatment Vs Day	16	3.149**	6.885**	7.671**	6.801**	3.781**	2.328**	18.204**
Repetition	3	1.829	0.650	0.51	0.968	0.450	3.105	0.865
CV (%)	-	12.44	64.66	37.98	17.02	9.61	40.43	14.00

N.S.- Not Significant; * - Significant at $P < 0.05$; ** - Significant at $P < 0.01$.

V.F. Variation factor; D.F. Degree of freedom; C.V. coefficient of variation.

Table 5: Results of the ANOVA for the effects of immersion with distilled water on the salic gleysol samples over time (days) and the two treatments.

VF	DF	Ca	K	P	Zn	Mn	Cu	Fe
		-----cmol/ dm ³ -----			-----mg/dm ³ -----			
Day	14	10.714**	8.096**	16.911**	6.478**	4.137**	7.792**	100.103**
Treatment	1	8.298**	0.252 ^{NS}	0.104 ^{NS}	0.818 ^{NS}	0.005 ^{NS}	12.142**	15.658**
Treatment Vs Day	12	-1.213 ^{NS}	5.750**	9.639**	1.074 ^{NS}	-0.501 ^{NS}	10.769**	13.586**
Repetition	3	1.793	0.683	0.422	1.067	1.241	1.130	1.216
CV (%)	-	12.44	36.71	26.34	28.08	39.77	47.44	10.19

N.S.- Not Significant; * - Significant at P < 0.05; ** - Significant at P < 0.01

V.F. Variation factor; D.F. Degree of freedom; C.V. coefficient of variation

The element phosphorus showed a significant difference between treatments in Haplic Gleysol. For Salic Gleysol this difference did not exist. The Tukey test indicated that the dry treatment provided significantly higher estimates of the phosphorus content, indicating that the availability of this element was affected by immersion. These modifications were due to the hydrolysis of the iron and aluminum phosphates, which liberated phosphorus in the exchange positions in the clays and oxy-hydroxides of iron and aluminum, as well as the reduction of Fe³⁺ to Fe²⁺, with the subsequent liberation of the phosphorus linked to these minerals (Fageria, 1984 [14]; Lima *et al.*, 2005 [22]), although the results of the experiment indicated that this did not occur uniformly in the two soils tested (Figure 2).

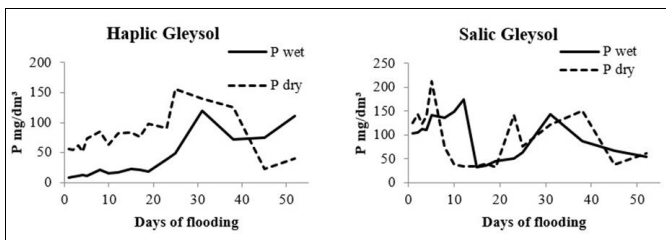


Fig 2: Variations in the concentration of phosphorus over time, according to the results of the two analytical procedures (humid and pre-dried soil)

In the case of the salic gleysol, which had the highest concentrations of phosphorus, the liberation of this element occurred at high levels at the beginning of the experiment, decreasing over time, but still remaining at relatively high levels. Given this, significant differences were not found between the two analytical procedures. The haplic gleysol presented relatively slow rates of increase of the phosphorus content, with the humid procedure detecting minor variations at the beginning of the process, as observed in previous studies (Silva *et al.*, 1988 [38], Lima *et al.*, 2005 [22]), but with both procedures identifying variations from the 30th day onwards, as well as a reduction in the concentration on the 45th day of immersion. Overall, then, the results of the analysis of the humid samples appear to represent the known behavior of this element in this type of environment, which starts with the initiation of the oxy-reduction processes, which influence the liberation of phosphorus at increasing rates during the first few days of immersion, as well as the anomalous pattern observed in the salic gleysol. It is important to remember that the high levels recorded for both types of substrate are a distinct characteristic of these soils. In Asia, Peng and Senadhira (1998) [27] found that flooded soils used for the cultivation of rice were mostly deficient in phosphorus and zinc.

In the case of iron, a micronutrient, the two treatments followed relatively similar patterns in the haplic gleysol, with larger

amounts being liberated in the pre-dried samples over the first 23 days, but subsequently a tendency to decrease, whereas in the humid soil treatment, a tendency to increase was observed (Figure 3). This confirms the tendency for iron concentrations to increase, reaching relatively high levels (Silva *et al.*, 1988; Lima *et al.*, 2005) [38, 22]. The ANOVA returned a significant difference (1%), with the Tukey test indicating that the humid sample was the better treatment for the analysis of the iron content. Iron suffers dynamic change in this environment (Schwertmann and Taylor, 1989) [31], reaching abnormally high levels in both soils and treatments, which may be toxic for some cultivated plants (Fageria, 1984) [14]. While both analytical procedures indicated a progressive increase in the availability of this element (Figure 3), although the analysis of the humid samples indicated slower rates of change at the beginning of the immersion period, a pattern recorded in previous studies (Ponnanperuma, 1972; Ferreira *et al.*, 1998) [28, 16]. This initial increase in the availability of iron was not detected with the same intensity in the analysis of the pre-dried samples. The salic gleysol presented a similar significant difference (1%) between treatments, with the Tukey test indicating the pre-dried sample as the best treatment. The variation in iron levels observed in the salic gleysol during the initial days of immersion in both treatments indicated higher values in the humid soil samples from the 31st day onwards.

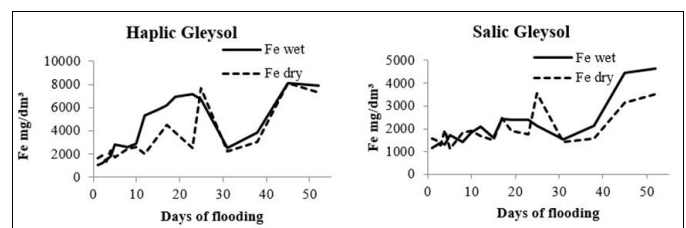


Fig 3: Variations in the concentration of iron over time, according to the results of the two analytical procedures (humid and pre-dried soil).

Manganese was affected directly by the modification in the environment, with a reduction in the first few days of immersion, when it was converted into the available form. The experiments indicate that this occurred in different ways in the two soils (Figure 4, Tables 2 and 3). In the haplic gleysol, concentrations varied from 151.5 g/dm³ (humid soil analysis) to 293.75 mg/dm³ (pre-dried soil) on the first day of immersion, reaching maximum values of 526 mg/dm³ by the 52nd day in the case of the humid sample, and 394.25 mg/dm³ by the 38th day for the pre-dried sample. Overall, then, relatively high values were recorded throughout the experiment in both types of analytical procedure. The ANOVA for the haplic gleysol indicated a significant difference between treatments, with the Tukey test confirming significantly higher values in the analysis of the pre-dried samples. In the case of this soil, however, the rapid reduction of the manganese was observed in both

treatments. Lower values were recorded for the humid soil samples at the beginning of the immersion period, although the difference between treatments disappeared from the fifth day of immersion onwards.

The concentrations of manganese in the salic gleysol were very low in comparison with the haplic soil, and decreased over the course of the study period. On the first day, the values were 14 mg/dm³ for the humid soil analysis and 9.25 mg/dm³ for the pre-dried samples, decreasing to 5 mg/dm³ by the 52nd day of immersion in both analytical procedures. Given this, the ANOVA found no significant difference between treatments for the salic gleysol.

Both analytical procedures confirmed high concentrations of manganese throughout the 52 days of immersion of the haplic gleysol, and low values in the salic gleysol. In the specific case of the haplic gleysol, however, the pattern presented by the analysis of the humid samples returned lower values, increasing rapidly over the first seven days, and then up to the 20th day of immersion, a period sufficient for the reduction of this element, with an increase in the concentration still observed on the 45th day. In the case of the salic gleysol, by contrast, similar low values were recorded throughout the study period by the two analytical procedures.

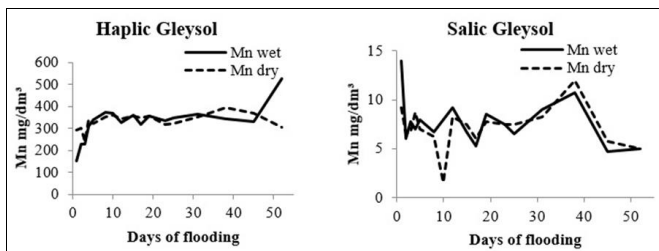


Fig 4: Variations in the concentration of manganese over time, according to the results of the two analytical procedures (humid and pre-dried soil).

Calcium was liberated at similar levels in both soils, with similar results being obtained in both treatments, although once again, lower levels were recorded in the humid soil samples over the first 10 days of immersion, in both types of soil (Figure 5). This may have occurred because the calcium had not yet been dissolved in the soil solution. There was no significant difference (ANOVA) in the levels of exchangeable calcium recorded in the different treatments for the haplic gleysol. Significant differences (1%) were recorded between treatments for the salic gleysol, however, with higher values being recorded for the pre-dried samples, which returned higher values in comparison with the humid soil samples from the 25th day of immersion onwards.

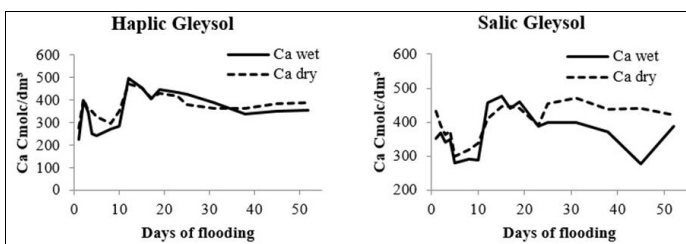


Fig 5: Variations in the concentration of calcium over time, according to the results of the two analytical procedures (humid and pre-dried soil)

Potassium levels in the salic gleysols were similar in the two treatments tested, with no significant difference found in the

ANOVA, whereas significant variation was recorded for the haplic gleysol. The liberation of the potassium can be observed at each stage in the analysis of the humid soil, although medium values were recorded from the pre-dried samples throughout the study period. While no significant differences were recorded in the case of the salic gleysols, the same pattern was recorded for the humid soil analyses. In this analysis, it was also possible to confirm stability in the exchange of cations from the 23rd day onwards in the haplic gleysol, and from the 31st day onwards in the case of the salic gleysol (Figure 6). The Tukey test indicated that this treatment was the most effective for the analysis of the haplic gleysol.

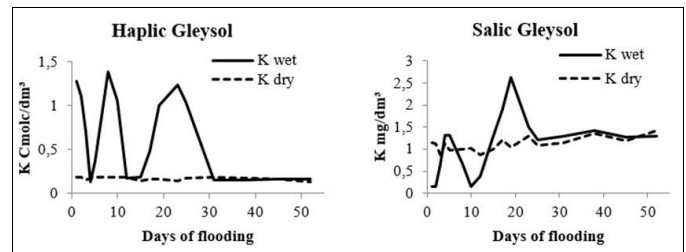


Fig 6: Variations in the concentration of potassium over time, according to the results of the two analytical procedures (humid and pre-dried soil).

Zinc and copper concentrations are not normally affected by the oxy-reduction reactions that occur typically in these soils (Fageria, 1984) [14], but they are altered indirectly by the migration to the soil solution occurring after the substantial production of other elements, such as iron and manganese, and the alteration of the pH, which reduces the availability of these elements at higher values, or even results in their fixation in neoformed minerals (Ponnamperuma, 1972 [28], Sanches, 1981 [29]; Fageria, 1984 [14]; Brinkman, 1985 [5]). However, while zinc did not become unavailable, it occurred at low concentrations, especially in the salic gleysol, in which it was liberated in reduced amounts, with a similar pattern being observed in the two treatments, which did not differ significantly (ANOVA). Higher values were recorded in the haplic gleysol, with a significant difference (1%) being recorded being treatments, with much higher values being recorded in the analysis of the humid samples (Figure 7, Tables 4 and 5).

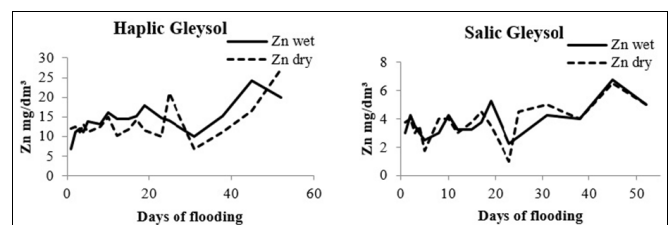


Fig 7: Variations in the concentration of zinc over time, according to the results of the two analytical procedures (humid and pre-dried soil).

Copper behaves in a similar way to potassium, increasing its contents, with these elements being liberated through discrete exchange events. In this closed system, with no external influence, copper should have decreased as pH values increased (Bertoni *et al.*, 1999; Mattar *et al.*, 2002; Silva, 2008) [2, 24, 35]. On the other hand, a small increase should have occurred due to production of other ions, such as those of iron and manganese, which may migrate to the soil solution. However, the concentration of this element continued to decrease over time, over a longer period than that analyzed here, but without its

complete elimination, given that the pH stabilized at approximately 6.5 in the haplic gleysol (Silva and Costa, 2011)^[37] and 5.5 in the salic gleysol (Silva and Costa, 2012)^[36].

In the haplic gleysol, variations in the concentration of copper over time were very similar in the two treatments (Figure 8). The concentrations of copper recorded in the humid samples ranged from 1 to 2.25 mg/dm³ up to the 25th day, increasing to 5 mg/dm³ by the 31st day of immersion, followed by a decrease. A similar pattern was observed in the results of the analysis of the pre-dried samples, with values of 1-3.25 mg/dm³ recorded prior to the 25th day, increasing subsequently to 6.25 mg/dm³. The Tukey test indicated significantly higher values for the dry samples, even though the humid samples indicated an increase in concentrations from the 45th day onwards, inverting the overall tendency recorded by both analytical procedures.

While the values recorded for the salic gleysol were very similar during the later part of the immersion period, significantly higher values (1%) were recorded overall in the analysis of the humid soil samples. Despite the similarities in the results, the analysis of the humid samples returned much higher values between the 19th and 23rd days, whereas the analysis of the pre-dried samples revealed much lower peaks on the 12th, and 23-25th days.

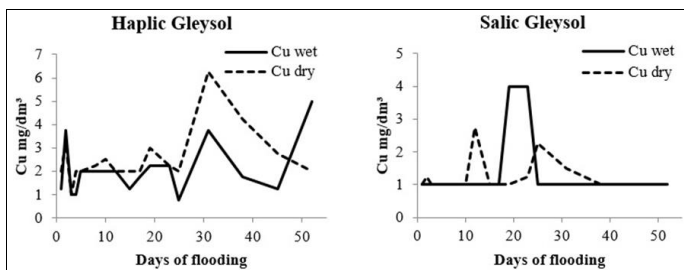


Fig 8: Variations in the concentration of copper over time, according to the results of the two analytical procedures (humid and pre-dried soil).

Significantly higher values were recorded for phosphorus, manganese, and copper in the analysis of the pre-dried samples of haplic gleysol, whereas iron, potassium, and zinc were more abundant in the humid samples. Calcium was the only element tested that did not vary significantly between treatments in the analysis of this soil type. A distinct pattern was recorded in the case of the salic gleysol, however, with no significant difference being found between treatments for phosphorus, potassium, manganese or zinc. Significantly higher values for iron and copper were recorded in the analysis of the humid samples, whereas calcium was higher in the pre-dried samples (Tukey test). This variation in the experimental response is important because it implies that specific responses in the fertility of the soil may be interpreted differently according to the analytical procedure employed.

Overall, then, while the analysis of the dried samples provided significantly higher values in some cases, suggesting more reliable results, the values recorded by the analysis of the humid samples were more similar to those reported in previous studies (Silva *et al.*, 1988; Lima *et al.*, 2005)^[38, 22]. This suggests that this approach is more sensitive to minor changes in the concentrations of the elements analyzed. Given this, the analysis of the humid samples appears to be a reliable approach to the assessment of the fertility of these inundated soils.

4. Conclusions

The analysis of the fertility of inundated soils based on the chemical assessment of the dried samples may produce results

that are invariably the same or closely similar. This may occur because the drying process reverts the characteristics of the samples to those of the aerobic soil, prior to reduction, altering the availability of the nutrients present in these substrates. As the fertility of the dry soil is not altered, the results will invariably be similar.

The present study indicates that this difference is not universal or consistent, given that some variation was observed in relation to the values recorded for the humid samples, with very similar concentrations being found in some cases. However, there were no absolutely divergent results between the two approaches. Where the two treatments produced values completely different in size, such as those recorded for manganese, both techniques recorded similar patterns of variation, in particular with regard to the differences between the types of gleysol, with relatively high concentrations being recorded for the haplic soil and lower values for the salic soil.

Both procedures also identified the same specific patterns of variation in the two types of soil analyzed. The liberation of elements by each soil type follows a distinct pattern related to its inherent characteristics, such as its vegetation and mineralogy. The values recorded for manganese are a good case in point. This element was present at high concentrations in the haplic gleysol, but occurred at very low concentrations in the salic gleysol, a result confirmed by both approaches.

Specific patterns of alterations to fertility were also recorded for each soil type. In particular, the analysis of calcium levels in the haplic gleysol did not vary significantly between treatments, while those for phosphorus, potassium, manganese, and zinc were also similar for the salic gleysol, independently of the procedure used. This indicates that the choice of procedure did not affect the results obtained for these elements.

Statistically, the analysis of the pre-dried samples returned significantly higher values for the elements phosphorus, manganese, and copper in the haplic gleysol, and for calcium, in the salic gleysol. However, the variation observed in the dried samples may have been more favorable, given the observation of higher values (Tukey test). In the case of manganese and phosphorus, however, the humid samples appeared to be more detailed, revealing subtle variation in the relatively low concentrations during the first few days of experimental immersion. The results from calcium and copper were similar, however, in both treatments, and were within the same fertility class.

The analysis of the humid soil samples appeared to provide more reliable results for the concentrations of potassium, zinc, and iron (in the haplic gleysol) and copper and iron (in the salic gleysol). Together with the results that found no significant difference between analytical procedures, this comparison indicates that the analysis of the humid samples was at least as reliable as that of the pre-dried samples in approximately 71% of the cases.

Overall, then, the analysis of the humid soil samples was considered to be satisfactory for the evaluation of the fertility of the haplic and salic gleysols tested under anaerobic conditions. The positive results and the lack of significant differences between procedures for many of the elements analyzed indicate that this approach can be used reliably. In particular, the analysis of the humid samples appears to be better for the identification of patterns of variation at relatively low concentrations. This supports the conclusions of the research, which indicate that soils subject to natural inundation present distinct characteristics in the field and the laboratory, following the drying process. These findings are especially important in the context of the

Amazon region, given the vast area covered by várzea soils.

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