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#### Scientific paper

### Abstract

Considering the soil as a controlling factor in the interaction of the ecotones, the study of the soil origin and formation becomes of great importance. Its mineralogical composition, as well as the attributes and properties related to these inorganic components are points that deserve prominence. Facing that, it was aimed with this work to analyze the soils of ecotones of

# Mineralogical, granulometrical, and chemical analysis, in soils of ecotones of the southwest of Tocantins

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the cerrado in the Ilha do Bananal in the state of Tocantins concerning its mineralogy, chemical and physical characteristics. Samples of TFSA were dispersed at 2.500 rpm for 3 minutes, removing the interfering agents (litung), the separation of the sand was accomplished through sieve of 0,053 mm and the fractions silt and clay, they were separated trough exhaustion of the fraction clay, being constituted in the fraction silt, which was sonicated, separating the fraction disaggregated clay (for siphonage) of the fraction silt. It used the dithionite and oxalate method to quantify the content of iron in the fraction clay and diffraction of ray-X for the characterization of the minerals. The mineralogical and chemical compositions of the soils have outstanding effect in the dispersion of the clay, which reflects in the fraction silt. In the chemical and mineralogical analysis, it verifies that the soils of that area are poor and with high weathering degree. There is high intensity of the minerals kaolinite or gibbsite in the fractions silt and clay. The studied classes possess low amount of dithinite iron, characterizing low mineral crystallisation.

Keywords: Mineralogy, X Ray, Rust of Crystalline and Amorphous Iron, Soil Classification, Fractioning.

# Introduction

Considering soil as a controller factor in the interaction of ecotones, the study of the soil origin and formation is of great importance. It, for its turn, has had great advances with mineralogy, aiming to know and characterize the different soil classes, originated by the multiple relations which are established between its formation factors. For JACKSON (1979), among them, in their mineralogical composition, as well as the attributed and properties related to these inorganic compounds are points which deserve emphasis.

Firstly, it is necessary to understand the mineralogical analysis of soil with the qualitative and quantitative recognition of the mineral constituents in the different texture fractions, i.e., sand, silt and clay. In a more general scope, the basic mineralogy itself has

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strong ties with the study of soils, since the methods used and knowledge produced over the crystalline structures of the minerals are very useful in the comprehension of the behavior of the primary and secondary minerals present in the soil.

In the same way, for VITORINO et al. (2003) it is necessary to recognize the validity and application of the mineralogical results in the different areas of Pedology, which denoted the strong ties of the soil mineralogy with the other areas of work, emphasizing the soil classification, soil management, soil fertility and plant nutrition, recovery of degraded areas, soil chemistry, etc.

Mineralogy is of great value due to the phenomenon of diffraction of x-rays, and it is possible to study materials in the atomic level, discovering and limiting their structure. From this, many other innovating contributions came from the most diverse studies, with emphasis in the possibility that Bragg provided of expressing the phenomenon in a quantitative way, as it will be discussed later. Thus, the present work aims to analyze soils of ecotones of the cerrado<sup>1</sup> for the Bananal Island in the state of Tocantins concerning its mineralogy, and chemical and physical characteristics.

### **Material and Methods**

The work was developed in the region of transition between the biomas cerrado and Ilha do Bananal. The sampling region is located between the municipalities of Araguaçu and Formoso do Araguaia. For the execution of the work, it was selected four soil profiles which represent the diversity of soil classes in the region. The region climate is of the type B2rA'a' – humid climate with small or null water deficiency, according to the method by THORNTHWAITE (1948) and an average rainfall of 1600 mm. After the classification, it was collected soil samples and geographic coordinates.

The selected samples were collected in natural conditions, being 3 samples, with the corresponding coordinates (UTM 22L, SAD69):

Profile 1: PLINTOSSOLO HÁPLICO Distrófico (FXd) 691800 and 8678817; Profile 2: CAMBISSOLO HÁPLICO Tb Distrófico (CXbd) 688371 and 8679822; Profile 3: LATOSSOLO VERMELHO-AMARELO Distrófico típico (LVAd) 680618 and 8684143 and Profile 4: GLEISSOLO HÁPLICO Tb Distrófico típico (GXbd) 671393 and 8690186.

For the chemical and physical characterization of the soils from each profile, samples were transported to the routine laboratory of the Universidade Federal de Viçosa (Federal University of Viçosa).

In the Department of Mineralogy the samples were identified, separated and placed to dry, and later it was passed the samples in a sieve with 2 mm (TFSA). Next it was taken samples of 10g of TFSA in duplicate, in tube of 50 mL. It was added 20 mL of water in each glass, and next it was added more 20 mL of NaOH 0.1 N trough manual shaking, and it was left to rest for 1 hour. Next, it was placed in high rotation agitation (250 rpm) for 30 minutes, and it was centrifuged and the supernatant was removed later. The next step was the addition of 25 mL of sodium hypochlorite at 10% in pH 9.5 per tube, for the soil dispersion, and then they are homogenized and placed in water bath at 75 - 80 °C for 30 minutes, and they were agitated each 5 minutes, and centrifuged (3.5 minutes at 2500 rpm) in the end of the 30 minutes, and the process was repeated two more times. Later, it was made a washing with NaCl 0.5 mol L-1 twice, discarding the supernatant material.

The samples without the organic matter contained in the tubes of 50 mL were passed in sieve of 0.053 mm for the removal of the fraction sand, and they were transferred, after being washed in running water, from the sieve to a becker of 600 mL with aid of a water jet, and placed in oven at 40 – 45 °C to dry for 72 hours.

The total content of clay was determined by the pipette method, according to the texture analysis proposed by EMBRAPA (1997). In the determination of the relation thin clay/thick clay, it was removed the iron oxides of 2 g of TFSA, trough five successive extractions of dithionite-citrate-bicarbonate (DCB) (MEHRA and JACKSON, 1973), performed in room temperature.

<sup>&</sup>lt;sup>1</sup> Brazilian region with characteristics of savanna

The dispersion was performed with solution of NaOH 0.01 mol L<sup>-1</sup>. Next, it was promoted the separation of the colloidal fraction, through successive siphoning and agitations. Thus, it was obtained the total deferrificated clay, which was separated in thick clay (between 2 and 0.2  $\mu$ m) and thin clay (smaller than 0.2  $\mu$ m), by centrifugation (JACKSON, 1979).

The identification of the compounds of the faction natural clay was performed by diffraction of X rays (XRD) in equipment Siemmens D-5000 with CoK $\alpha$  radiation, graphite monochromator and it was operated at 40 kV and 25 mA. Samples were crushed in agate mortar, together with 5% of NaCl (Merck ACS, ISO PA), used as internal pattern. The set was built in a glass support, after soft pressure of the sample over rough paper, so that it would minimize the preferential orientation of particles. The irradiations ranged from 10 to 70° 2 $\theta$ , with interval of 0.02° 2 $\theta$  for each 6s. It is important to emphasize that it was maintained the same support for all the evaluated samples, aiming to avoid possible changes of the spectrums (peak length or intensity) related to changes in the sample-holder.

As a result of the submission of one sample to an equipment of diffraction of x rays, there was a x and y data set which correspond, respectively, to the  $2\theta$  degrees and the intensity (number of times that the radiation was detercted), whose interpolation generates a graphic known as X-Ray diffractogram.

# **Results and discussion**

In Table 1, it can be verified increasing percentage of clay with the increase of depth. For thick clay, it is observed converse effect to the percentage of silt and thin sand remained constant in all the profiles.

 Table 1. Estimate of soil fractions in PLINTOSSOLO HÁPLICO Distrófico (FXd) horizons, Formoso do Araguaia - TO, 2007.

Horizon	Clay (%)	Silt (%)	Thin Sand (%)	Thick Sand (%)	Silt/Clay
А	35	4	15	46	0.12
AB	42	4	15	39	0.1
Btf	59	4	14	23	0.07

The profile presents B horizon texture, where there is transport of clay from the A horizon to the B horizon and according to morphological discussion there is formation of plinthite in the characteristic horizon of approximately 30%, being thus characterized as Btf (EMBRAPA, 2006). It has texture from clayey-sand to clay.

 Table 2.
 Chemical analysis of PLINTOSSOLO HÁPLICO Distrófico (FXd), Formoso do Araguaia - TO, 2007.

Uorizon	pН	Р	Κ	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Al <sup>3+</sup>	H+Al	SB	Т	V	MO	P-rem
110112011	$(H_2O)$	mg o	dm-3		•••••	cmol <sub>c</sub>	dm-3	••••	••••••	%	dag kg-1	mg L-2
А	5.25	0.4	10	0.04	0.02	0.05	2.40	0.10	2.50	4.0	1.48	23.30
AB	5.30	0.3	5	0.11	0.05	0.00	2.20	0.17	2.37	7.2	1.34	8.70
Btf	5.44	0.3	9	0.06	0.01	0.00	1.70	0.09	1.79	5.0	0.40	4.00

In Table 2 it is observed pH with mean acidity and low base saturation, characteristic of this soil class. In the relation Feo/Fed, (Table 3), it is noticed degree of crystallinity, since normally values of the relation Feo/Fed inferior to the unit characterize forms of crystal iron (KAMPF and DICK, 1984).

**Table 3.** Content of iron oxides of PLINTOSSOLO HÁPLICO Distrófico (FXd) horizon, from the fraction Clay, through the methods oxalate (Feo) and citrate-dithionite (Fed) and its Feo/Fed relation.

Horizon	Oxalate	Dithionite	Feo/Fed
А	1.688	7.811	0.216
AB	1.469	5.824	0.252
Btf	-	8.231	-

The diffractograms of the fractions sand, silt and clay of the A, AB and B horizons of the profile 1, PLINTOSSOLO HÁPLICO Distrófico, present mineralogy in the fraction sand constant concerning the contents of quartz (Figures 1, 2 and 3). This was already expected, since in the fraction sand, quartz (SiO<sub>2</sub>) is the dominant material.

In the fraction silt of the horizons A, AB and B, minerals with highest intensity were bayerite (Al (OH)<sub>3</sub>, plagioclase ((Ca,Na) Al(Al,Si) Si<sub>2</sub>O<sub>8</sub>) and gibbsite  $(Al(OH)_{3})$ respectively. Even though gibbsite and kaolinite may occur in the fraction silt, the presence of these minerals, as it was evidenced in the diffractograms, may also indicate that the level of energy used in the process of separation was not enough for the complete removal of these minerals (VITORINO et al., 2003). The permanence of the peaks of gibbsite in the diffractograms of silt, reinforces the role of this mineral in the higher resistance that the aggregated offer to the dispersion (NETTO, 1996 and FERREIRA et al., 1999 and RESENDE et al., 1999); they also report that the presence of gibbsite helps to maintain the microgranular structure.

For the fractions of Clay (Figures 1, 2 and 3), the highest intensities are observed in the horizons A, AB and B for siderotil ((Fe,Cu) SO<sub>4</sub>.5H<sub>2</sub>O) and birnessite (Na<sub>4</sub>Mg<sub>14</sub>O<sub>27</sub>.9H<sub>2</sub>O), laumontite (Ca(Si<sub>4</sub>Al<sub>2</sub>)O<sub>12</sub>.4H<sub>2</sub>O) and bayerite, and sauconite (Na<sub>0.3</sub>Zn<sub>3</sub>(Si,Al)<sub>4</sub>O<sub>10</sub>(OH).4H<sub>2</sub>O), basaluminite (Al<sub>2</sub>(OH)<sub>10</sub>SO<sub>4</sub>.5H<sub>2</sub>O) and anhydrite (CaSO<sub>4</sub>), respectively. In this fraction, the degree of crystallinity is higher in relation to sand and silt.

When evaluating the granulometry of CAMBISSOLO HÁPLICO Tb Distrófico horizons, it is verified higher percentage of clay in the Bi, C and A horizons, respectively. Presenting texture which ranges between sandy-clay-loam to clay, massive structure and the average relation between silt and clay is 0.61, higher than 0.6, which is the minimum indicated for this soil class, characterizing one Bi (Table 4).

In presents in the fraction sand the mineral muscovite (Figure 4), according to EMBRAPA (1999), presents 6% or more of muscovite in the fraction total sand. In the classification it presented more than 5% of the soil volume of gravels.

**Table 4.** Soil texture fractions in CAMBISSOLO HÁPLICO Tb Distrófico (CXbd) horizons, Formoso doAraguaia - TO, 2007.

Horizon	Clay (%)	Silt (%)	Thin Sand (%)	Thick Sand (%)	Silt/Clay
А	28	16	26	30	0.57
Bi	45	16	16	23	0.36
С	37	33	19	11	0.89

In the chemical analysis (Table 5) it is observed low base saturation, which is characteristic for this soil class, since, according to EMBRAPA (2006), for this kind of class, the base saturation ranges from high to low.

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**Table 5.** Chemical analysis of CAMBISSOLO HÁPLICO Tb Distrófico (CXbd) formation, Formoso doAraguaia - TO, 2007.

Uorizon	pН	Р	K	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Al <sup>3+</sup>	H+Al	SB	Т	V	MO	P-rem
110112011	$(H_2O)$	mg o	dm-3	••••••	••••••	cmol	c dm-3		••••	%	dag kg-1	mg L-2
А	5.45	0.7	56	0.79	0.92	0.10	3.70	1.85	5.55	33.3	2.96	30.40
Bi	5.92	0.3	28	0.40	1.06	0.00	1.70	1.53	3.23	47.4	1.34	14.20
С	5.80	0.3	2	0.01	0.00	0.72	3.20	0.02	3.22	0.6	0.81	19.70

It presents high content of Feo/Fed in the Bi horizon (Table 6), with highest degree of crystallinity, in relation to the other soil profiles which were analyzed, thus it can be noticed highest weathering in the Bi horizon. This relation measures qualitative index of the degree of crystallinity of the oxides which compound the fraction clay (KÄMPF, 1988).

**Table 6.** Content of iron oxide in CAMBISSOLO HÁPLICO Tb Distrófico (CXbd) horizons, from the Clay fraction, through the methods oxalate (Feo) and citrate-diothinite (Fed) and its relation Feo/Fed.

Horizon	Oxalate	Dithionite	Feo/Fed
	Fe g		
A	7.772	17.372	0.447
Bi	5.606	6.902	0.812
С	2.280	5.521	0.413

By observing the factions sand, silt and clay, it can be named the peaks with highest intensity in the fraction sand quartz (SiO<sub>2</sub>) (figures 4, 5 and 6), in the fraction silt it is noteworthy laumontite (Ca(Si<sub>4</sub>Al<sub>2</sub>)O<sub>12</sub>.4H<sub>2</sub>O), kaolinite (Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>) and fibroferrite  $(Fe^{+3}(SO_4)(OH).5H_2O)$ , in the horizons A, Bi and C. The Cambissolo in its horizon "Bi" present intense reflexes of kaolinite (VITORINO et al., 2003). In the fractions clay the lowest peak identified in the horizons A, Bi and C were phillipsite ((K, Na, Ca)<sub>2</sub>(Si,Al)<sub>8</sub>O<sub>16</sub>.6H<sub>2</sub>O) and kaolinite, lepidocrocite (Fe<sup>+3</sup>O(OH) and kaolinite, and psilomelane  $(BaMn^{2+}Mn_{4}+O_{16}(OH)_{4})$  and greigite  $(Fe_{3}S_{4})$ , respectively.

This is probably associated to the fact that these soils were developed over a preweathered material, according to reports by RESENDE et al. (1988) that some CAMBISSOLOS may result from the accented removal of materal of ancient LATOSSOLOS, formed from the C horizon subjacent to the LATOSSOLOS itself.

In this case, in the horizon characteristic of the current CAMBISSOLO, there is the same mineral which dominates the silt fraction, kaolinite.

The intense reflex of kaolinite in the silt fraction of the CAMBISSOLOS may also be related to the presence of pseudomorphs of kaolinite with silt size (PINTO, 1971).

Analyzing the LATOSSOLO VERMELHO-AMARELO Distrófico típico (Table 7), it can be verified increase in the percentage of clay with the increase of depth and low percentage of silt, texture sandy-clayloam and clayey-sand, in the diagnoses horizon it was grain structure, and it has relation silt clay lower than 0.7 in the B horizon.

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**Table 7.** Estimate of the soil fractions in LATOSSOLO VERMELHO-AMARELO Distrófico típico (LVAd)horizons, Formoso do Araguaia - TO, 2007.

Horizon	Clay (%)	Silt (%)	Thin Sand (%)	Thick Sand (%)	Silt/Clay
А	28	4	42	26	0.14
В	39	6	36	19	0.15

In the chemical analysis according low cation exchange capacity and pH with presented in the Table 8, it is possible verify a medium acidity.

 Table 8. Chemical analysis of the Latossolo VERMELHO-AMARELO DISTRÓFICO típico (LVAd), Formoso do Araguaia - TO, 2007.

Uorizon	pН	Р	K	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Al <sup>3+</sup>	H+Al	SB	Т	V	MO	P-rem
110112011	$(H_2O)$	mg d	lm-3		• • • • • • • • • • • •	cmol	dm-3	•••••	••••	%	dag kg-1	mg L-2
А	4.70	0.3	8	0.04	0.01	0.24	3.00	0.07	3.07	2.3	1.07	27.00
В	5.22	0.2	3	0.06	0.06	0.00	1.60	0.13	1.73	7.5	1.07	17.00

In the relation Feo/Fed according presented in the Table 9, it is observed low

degree of crystallinity and in the superficial horizon there is lower weathering..

**Tabela 9.** Content of iron oxides in the LATOSSOLO VERMELHO-AMARELO Distrófico típico (LVAd), from the Clay formation, trough the methods oxalate (Feo) and citrate-dithionite (Fed) and their relation Feo/Fed.

Horizont	Oxalate	Dithionite	Feo/Fed
Homzoni	Fe ş		
А	2.478	7.844	0.316
В	2.091	5.319	0.393

In the process of fractioning, it is possible to see that in the horizons A and B of this Latossolo, quartz ( $SiO_2$ ) is the predominant mineral in the sand fraction (Figures 7 and 8).

In the fraction silt there is higher predominance of sauconite  $(Na_{0,3}Zn_3(Si,Al)_4O_{10}(OH)_2.4H_2O)$  and coquimbite  $(Fe^{+3}_2(SO_4)_3.9H_2O)$  in the horizons A and B, respectively. For the fraction clay in the same horizons respectively, it can be observed sauconite and greigite  $(Fe_3S_4)$ , and kaolinite  $(Al_2Si_2O_5(OH)_4)$ . The presence of kaolinite in the

B horizon is characteristic in this class, in which it is dominant in the clay fraction (GREGRORICH et al, 1988).

It is observed in Gleissolo Háplico Tb Distrófico típico horizons (table 10), trasnport of Clay from the horizon A to the horizon Cg and texture sandy-loam to clays-sandy-loam. The gray color may be given by the reduced iron,  $Fe^{2+}$ , or by the absence of oxidized Fe,  $Fe^{3+}$ , which gives to the soil a gleyey aspect (RESENDE, 1990).

**Table 10.** Estimate of the soil fractions in GLEISSOLO HÁPLICO Tb Distrófico típico (GXbd) horizons, Formoso do Araguaia - TO, 2007.

Horizon	Clay (%)	Silt (%)	Thin Sand (%)	Thick Sand (%)	Silt/Clay
А	13	6	53	28	0.46
Cg	27	7	40	26	0.26

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**Figure 7** – Difractogram of x-rays in the A horizon of the Profile 3 (Cambissolo Háplico Tb Distrófico) from ecotones, Formoso do Araguaia – TO, 2007. (a) sand, (b) silt and (c) Clay. Qz: quartz; Bt: bayerite; Gr: greigite; St: sauconite; Cl: carnalitte; Dp: diaspore; Cb: cristibalite; Gb: gibbsite.

**Figure 8** – Difractogram of x-rays in the B horizon of the Profile 3 (Cambissolo Háplico Tb Distrófico) from ecotones, Formoso do Araguaia – TO, 2007. (a) sand, (b) silt and (c) Clay. Qz: quartz; Dp: diaspore; Ct: kaolinite; Vs: variscite ; Cq: coquimbite; Vt: vermiculite; Vr: valerite; As: anatase.

In the analytical characterization of soil, according data present in the Table 11, it

can be verified a very high acidity in this soil a and low base saturation.

**Table 11.** Chemical analysis of one GLEISSOLO HÁPLICO Tb Distrófico típico (GXbd), Formoso do Araguaia - TO, 2007.

Horizon	pН	Р	Κ	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Al <sup>3+</sup>	H+Al	SB	Т	V	MO	P-rem
Horizon	$(H_2O)$	mg d	lm-3		•••••	cmol	c dm-3		••••	%	dag kg-1	mg L-2
А	4.36	0.3	3	0.02	0.00	0.29	2.20	0.03	2.23	1.3	0.94	41.80
Cg	4.62	0.3	4	0.02	0.00	0.43	2.70	0.03	2.73	1.1	0.94	23.00

In the contents of iron oxide (Table 12), it is observed that the highest content of iron extracted in the fraction was found in A horizon, and it can be seen that there is higher amount of crystalline iron in relation to the amorphous in the relation Feo/Fed, characterizing low weathering, describing the hidromorphous soil class.

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**Table 12.** Content of iron oxide in the GLEISSOLO HÁPLICO Tb Distrófico típico (GXbd) horizons, from the fraction Clay, trough the methods oxalate (Feo) and citrate-dithionite (Fed) and its Feo/Fed relation.

Harizon	Oxalate	Dithionite	Feo/Fed
Horizon	Fe g		
А	2.139	23.196	0.092
Cg	1.026	6.228	0.165

In the fraction sand of the presented horizons (Figures 9 and 10), it is noteworthy the presence of quartz (SiO<sub>2</sub>), which was already expected, since it is the most abundant and most frequent mineral of the eruptive, sedimentary or metamorphic rocks (FONT\_ALTABA and SAN MIGUEL, 1980). In the fraction silt it is observed the presence of quartz in the horizon A and kieserite (MgSO<sub>4</sub>.H<sub>2</sub>O) in the horizon B, the quartz presented is due to the higher difficulty of individualization of the particles of this soil during the dispersion (VITORINO et al, 2003). For the fraction clay, the highest frequencies observed were for antigorite ((Mg,Fe<sup>+</sup>)<sub>3</sub>(Si<sub>2</sub>O<sub>5</sub>)(OH)<sub>4</sub>) in lower soil depth and nacrita (Al<sub>4</sub>[(OH)<sub>8</sub>] Si<sub>4</sub>O<sub>10</sub>]) in a greater soil depth.





**Figure 9** – Difractogram of x-rays in the A horizon of the Profile 7 (Gleissolo Háplico Tb Distrófico típico) from ecotones, Formoso do Araguaia – TO, 2007. (a) sand, (b) silt and (c) Clay. Qz: quartz; Dp: diaspore; Bk: brookite; Bt: biotite; Bs: birnessite; Vs: variscite; Es: smectite; Ag: antigorite; Bt: bayerite.

**Figure 10** – Difractogram of x-rays in the C horizon of the Profile 7 (Gleissolo Háplico Tb Distrófico típico) from ecotones, Formoso do Araguaia – TO, 2007. (a) sand, (b) silt and (c) Clay. Qz: quartz; Ft: feldspars-k; Bk: brookite; Bt: biotite; Bs: birnessite; Vs: variscite; Es: smectite; Kt: kieserite; Nt: nacrita.

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# Conclusions

The mineralogical and chemical compositions of soils have marked effect in the clay dispersion, with reflexes in the silt fraction.

In the analytical character, it is verified that the soils of this region are poor and with high degree of weathering. There is higher intensity of minerals kaolinie or gibbsite in the fractions silt and Clay. The studied classes have low quantity of

dithionite iron, characterizing low mineral crystallisation.

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