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Chemical, mineralogical and geotechnical characterization of the barreiras formation and post-barreiras sediments – southern coast of Rio Grande do Norte state, Brazil

Caracterização química, mineralógica e geotécnica de sedimentos da formação barreiras e pós-barreiras - litoral sul do Rio Grande do Norte, Brasil

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Abstract: The Barreiras Formation, of sedimentary origin (Miocene), extends along the Brazilian coast and has been studied by several researchers due to its extension and spatial variability, in addition to being in a densely populated region of high economic interest. This formation is overlain by Quaternary sediments, the so-called Post-Barreiras. The aim of this paper was to study the chemical, mineralogical and geotechnical properties of the Barreiras Formation and Post-Barreiras sediments, located on the southern coast of Rio Grande do Norte state. The sediments were collected from different locations, and x-ray fluorescence, atomic absorption spectrophotometry, x-ray diffraction and scanning electron microscopy tests were performed. Geotechnical properties were determined by particle size analysis, liquid and plastic limits and the specific gravity of solids. Chemical analysis showed silicon, aluminum and iron as the main chemical compounds. The Barreiras Formation sediments exhibited high iron oxide concentrations and the presence of quartz, kaolinite, hematite and goethite minerals. On the other hand, quartz, nacrite, anatase, and a low iron oxide content were identified for Post-Barrier sediments. From the geotechnical point of view, the sediments were classified as SC or SC-SM with low to medium plasticity.

Keywords: Barreiras Formation; Mineralogy; Geotechnical Characterization.

Resumo: A Formação Barreiras, de origem sedimentar (idade Miocênica), se estende ao longo do litoral brasileiro e tem sido objeto de diversas pesquisas, devido sua extensão e variabilidade espacial, além de se encontrar numa região intensamente povoada e de elevado interesse econômico. Sobre ela encontra-se uma camada de sedimentos do Quaternário, denominados de Pós-Barreiras. O objetivo deste trabalho é o estudo das propriedades químicas, mineralógicas e geotécnicas de sedimentos da Formação Barreiras e Pós-Barreiras, localizadas no litoral sul do Rio Grande do Norte. Foram coletadas amostras de diferentes pontos para a realização de ensaios de fluorescência de raios-x, espectrofotometria de absorção atômica, difração de raios-x e análises através do microscópio eletrônico de varredura. As propriedades geotécnicas foram determinadas a partir de ensaios de granulometria, limites de liquidez e plasticidade e massa específica dos sólidos. Os resultados das análises químicas apresentaram sílicio, alumínio e ferro como principais elementos químicos. Os sedimentos da Formação Barreiras apresentaram elevadas concentrações de ferro e ocorrência dos minerais quartzo, caulinita, hematita e goetita. Foram classificados como SC ou SM. Por outro lado, os sedimentos Pós-Barreiras apresentaram quartzo, nacrita e anatásio, e baixo teor de óxido de ferro. Sob o ponto de vista geotécnico, os sedimentos foram classificados como SC ou SC-SM de baixa a média plasticidade.

Palavras-chave: Formação Barreiras; Mineralogia; Carcterização Geotécnica

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1. Introduction

Barreiras Formation deposits are found along the Brazilian coast from the states of Amapá to Rio de Janeiro. This formation fills sedimentary basins that originated during the separation of the African and South American contents (Rossetti *et al.*, 2013). The top of the formation contains lateritic soil, covered in places by Post-Barreiras sediments, which encompass a large portion of the coastal Quaternary of Brazil (Bezerra *et al.*, 2006).

The Barreiras Formation has attracted the attention of researchers for its age, origin and geotechnical properties. A detailed study of this unit is important in the fields of engineering and economics, given that a large number of infrastructure projects in Rio Grande do Norte state (RN) have been built on this formation. In addition, there is intense use of its sediments as deposits for earthworks. Thus, it is essential to perform a detailed geological, mineralogical and geotechnical study, in support of engineering projects. To that end, researchers have studied the properties of Barreiras Formation sediments in RN (Santos Jr. *et al.*, 2015; Lucena 2015; Souza, 2016; Barbosa, 2017; Taquez, 2017; Sousa, 2018). However, given the size and importance of this formation for the region, there is a need for complementary studies. In addition, there is a gap in characterizing Post-Barreiras sediments. Thus, the aim of the present study was to investigate different areas of the southern coast of Rio Grande do Norte, including Barreira Formation sediments and Post-Barreiras surface sediments, and determine the chemical, mineralogical and geotechnical properties.

2. General and regional aspects of the barreiras formation

The Barreiras Formation was initially deposited in the middle Miocene, when sea levels rose. The highest part of the formation formed in the last marine transgression and regression episode during the same period (Rossetti *et al.*, 2013). This sedimentation process of the Miocene ended with the fall in sea levels. According to Rossetti *et al.* (2013), at this time a lateritic soil horizon formed on the top of Barreiras, lasting until the Quaternary. During this period, most of the Brazilian coast remained without sediment deposits, exposed to subarea erosion (wind and rain) and the development of this lateritic layer, composed of ferruginous concretions and rounded quartz grains. After this horizon was formed, the sediments known as Post-Barreiras were deposited.

Some authors consider that the origin of the Barreiras Formation is predominantly continental (Vilas Bôas *et al.*, 2001; Lima *et al.*, 2006;). However, in the equatorial portion of the formation, there are records of depositional environments influenced by marine processes and marine transition environments. In the eastern portion, where there is a clearer picture of the continental origin, there is also evidence of sediment deposition with marine influence, including in the Potiguar and Paraíba sedimentary basins (Rossetti *et al.*, 2012; Rossetti *et al.*, 2013).

The study regions of the present study are located on the east coast of Rio Grande do Norte state (RN) in Northeastern Brazil (Figure 1), encompassing sediments from both the Barreiras Formation and Post-Barreiras sediments. In geomorphological terms, the region is characterized by the presence of tablelands (Barreto *et al.*, 2004), and in some stretches of the coast the tablelands come into contact with the sea to form cliffs (Santos Jr. *et al*., 2015).

Figures 2a-c present the typical profile of the sediment layer of Barreiras Formation, covered by a layer of Quaternary sediments, denominated Post-Barreiras. These two horizons are divided by an uncomformity marked by the presence of a well-developed lateritic soil horizon of varied colors, with thickness varying between 3 and 10 m.

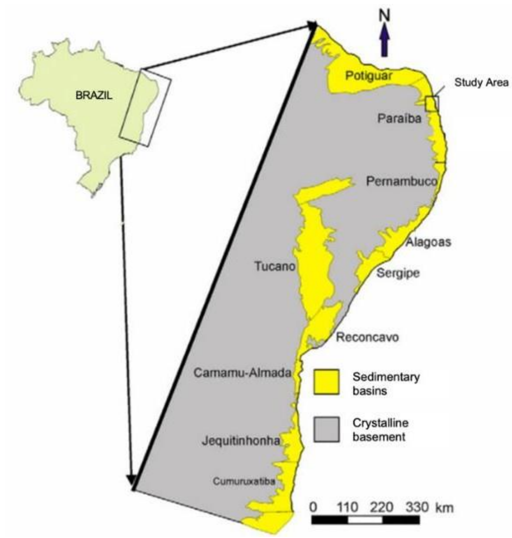
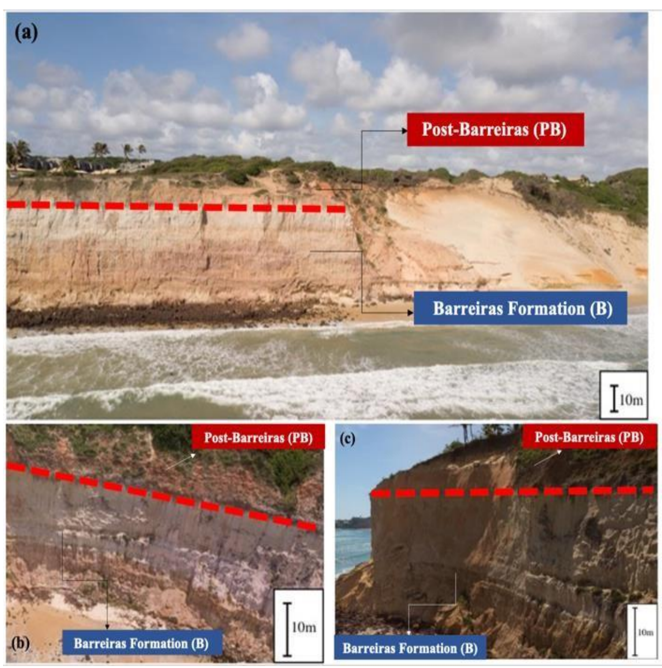


Figure 1 – Sedimentary basins on the east coast of Brazil and study location.
Source: Adapted from Rossetti et al. (2013).

A joint review of studies with geotechnical analysis of Barreiras Formation sediments in RN was conducted by Santos Jr. *et al.* (2015). The materials were classified in the Unified Soil Classification System (USCS), primarily as clayey sand (CS). All the samples exhibited a liquid limit of less than 50% and maximum plasticity index of 20%. The graphs of Figures 3 and 4 illustrate the plasticity chart and clay fraction activity of the samples studied. The data were updated to include more recent studies, such as those by Lucena (2015), Souza (2016), Barbosa (2017), Taquez (2017) and Sousa (2018).



Figures 2a-c – Typical profiles of cliffs along the east coast of Rio Grande do Norte, exhibiting the horizons of Barreiras Formation and Post-Barreiras sediments.
Source: Authors, 2020.

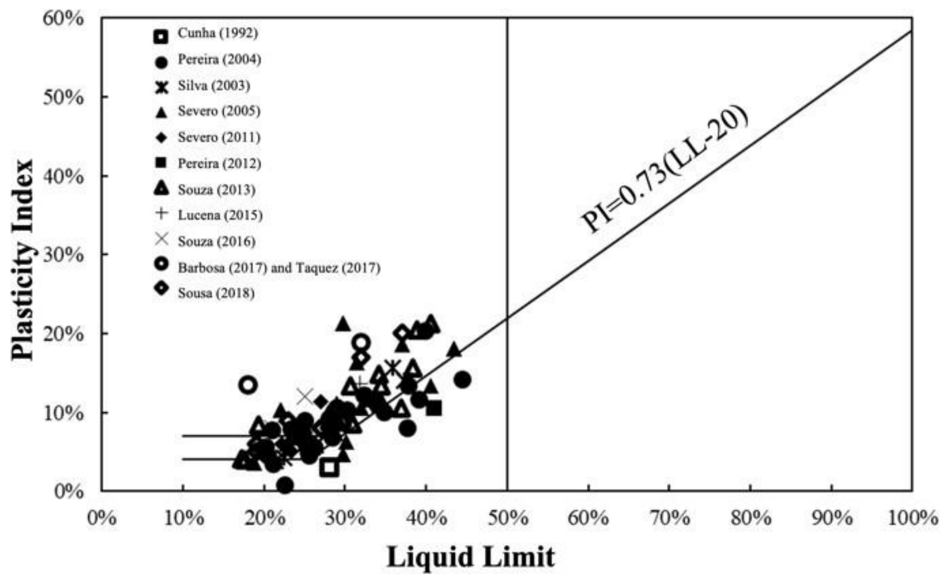


Figure 3 – Plasticity chart of Barreiras Formation soils in Rio Grande do Norte state.
Source: Adaped from Santos Jr. et al. (2015).

3. Materials and methods

The experimental program consisted of defining the areas along the south coast of RN where the Barreiras Formation (B1, B2 and B3) and Post-Barreiras sediments (PB0, PB1, PB2, PB3 and PB4) are found. Samples B1, B2 and B3 were collected in excavations of deposits or borrow pits that supplied the materials used for road embankments. Samples PB0 to PB4 were removed from the surfaces of cut slopes along highway BR-101. The geographic coordinates of the collection sites are presented in Table 1 and illustrated in Figure 5.

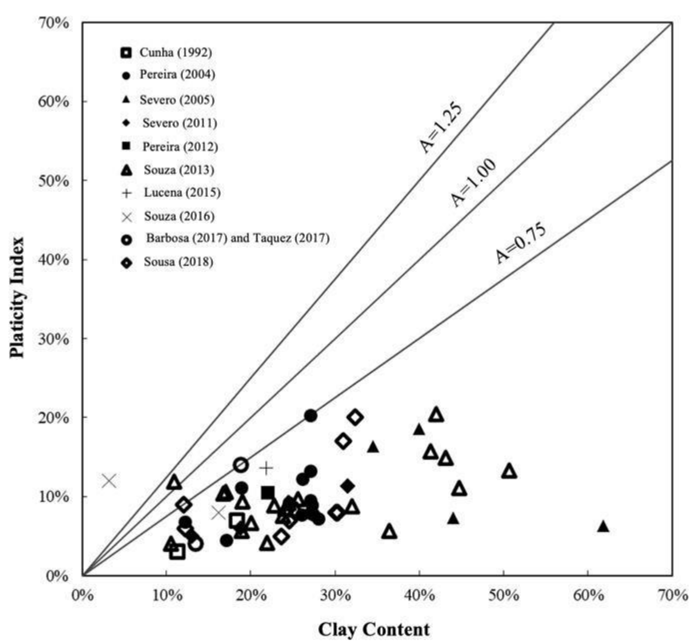


Figure 4 – Clay fraction activity of Barreiras Formation soils in Rio Grande do Norte.
Source: Adapted from Santos Jr. et al. (2015).

Table 1 – Geographic coordinates of the sample collection points.

Sample	Latitude	Longitude
B1	6°05'15''S	35°14'02''O
B2	6°13'47''S	35°08'38''O
B3	6°30'15''S	35°06'40''O
PB0	5°45'41''S	35°13'27''O
PB1	5°57'25''S	35°15'54''O
PB2	6°07'10''S	35°13'46''O
PB3	6°07'26''S	35°13'45''O
PB4	6°16'45''S	35°12'19''O

Source: Authors, 2020

3.1. Barreiras Formation Samples (B1, B2 and B3)

Chemical and mineralogical analysis of samples B1, B2 and B3 consisted of dividing the materials collected into two fractions, denominated concretion and sediment when larger and smaller than 9.5 mm, respectively. Sediment and concretion were differentiated in order to conduct an in-depth study of ferruginous concretions that formed in latertitic materials belonging to the Barreiras Formation of RN.

Later, the fractions were divided into subfractions, according to Table 2. The separation of the sediment fraction into subfractions consisted of wet mechanical sieving. In this process, materials with a diameter of more than 0.075 mm undergo deflocculation and sieving with running water. Sedimentation was performed to separate the materials with a diameter smaller than 0.075 mm. The concretion fraction, in turn, was immersed in water for four days and then disaggregated by impact. The material was then broken up and submitted to sedimentation, obtaining two subfractions (Table 2).

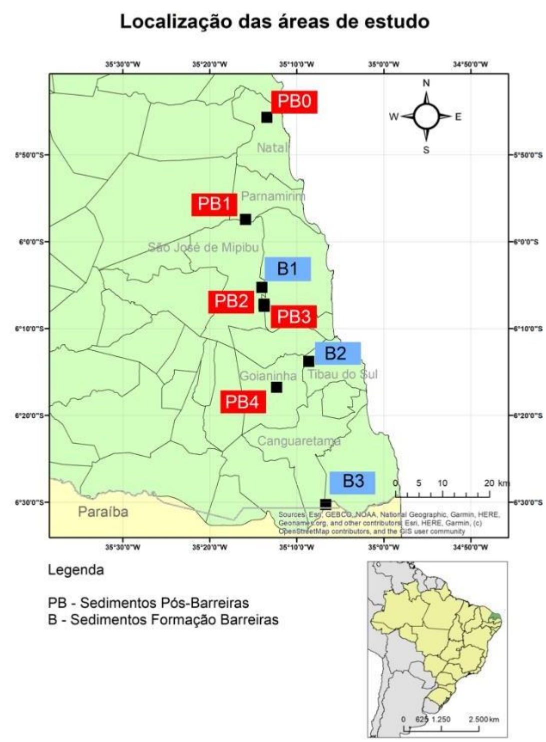


Figure 5 – Sample collection points along the south coast of Rio Grande do Norte.
Source: Authors, 2020.

All the subfractions underwent chemical analysis using atomic absorption spectrophotometry (AAS) and X-ray diffraction (XRD). The concretion microstructure was observed with a Stereoscan Mark 2 scanning electron microscope (SEM) (Cambridge Instrument Company).

The geotechnical characterization tests followed NBR 6457 guidelines (2016). Preparartion was preceded by air drying the sample, followed by disaggregation and homogenization. Particle size analyses were conducted in line with NBR 7181 (ABNT, 2016), specific gravity of grains according to NBR 6508 (ABNT, 1984), liquid limit (LL) and plastic limit (PL) in accordance with NBR 6459 (ABNT, 2016) and NBR 7180 (ABNT, 2016), respectively.

Table 2 – Fractions and subfractions of sediments and tests conducted with Barreiras (B) and Post-Barreiras (PB) formation

Fraction	**Subfraction (mm)**	**Tests**		
		Chemical Analysis	**XRD**	**SEM**
Concretion	0.075 - 0.002	X	X	X
	< 0.002	X	X	
Sediment	9.5 - 2.00	X	X	-
	2.00 - 0.075	X	X	-
	0.075 - 0.002	X	X	-
	< 0.002	X	X	-

Source: Authors, 2020.

3.2. Post-Barreiras Samples (PB0, PB1, PB2, PB3 and PB4)

Sample PB0 was initially separated into sand and clay fractions and then submitted to XRF and XRD tests as well as scanning electron microscopy (SEM), using a Shimadzu SSX-550 microscope. Particle size separation was performed to identify the chemical and mineralogical components of each fraction. Initially, a 0.075 mm sieve was used to separate the coarse from fine material. The sand retained was washed and then dried in an oven. The fine material that passed though the sieve was separated and added with a solution of sodium hexametaphosphate. This solution was left to rest for 24 hours, submitted to mechanical agitation, poured into a beaker and then left to rest again for a further 24 hours. The sedimentation method, based on Stoke's law, was applied. The upper part of this suspension was removed and dried in an oven, obtaining the clay fraction.

Samples PB1 to PB4 were submitted to semiquantitative x-ray fluorescence analysis with loss on ignition (XRF), using a Shimadzu EDX-720 energy dispersive x-ray fluorescence spectrometer and X-ray diffraction using a Shimadzu XRD-6000 x-ray diffractometer. These analyses were conducted with whole samples, without separating the sand and clay fractions

The geotechnical characterization tests for the PB sample followed the same guidelines and procedures used in the Barreiras Formation tests described in item 3.1.

4. Characterization of barreiras formation sediments (b)

4.1. Chemical and mineralogical analysis of samples B1, B2 and B3

The chemical analysis results of samples B1, B2 and B3 are presented in Tables 3, 4 and 5, respectively. The main oxides identified were silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3). The table also shows the silica/sesquioxide ratio (Kr). According to Winterkorn and Fang (1991), slateritic soils exhibit Kr<2 for the clay fraction. All the subfractions with diameter smaller than 0.002mm obtained Kr<2.

Table 2 – XRF test results of sample B1 expressed in %.

Material	**B1-S**				**B1-C**	
ϕ (mm)	**I (%)**	**II (%)**	**III (%)**	**IV (%)**	**V (%)**	**VI (%)**
SiO_2	77.40	91.26	51.50	31.40	51.81	8.74

Fe_2O_3	11.58	2.00	8.00	6.20	37.15	34.77
Al_2O_3	6.00	4.41	37.20	30.71	3.62	15.10
TiO_2	0.29	0.11	0.62	0.69	0.67	0.28
MnO	0.02	0.01	0.01	0.01	0.01	0.01
CaO	0.85	0.29	0.05	0.08	0.05	0.09
MgO	0.06	0.02	0.07	0.12	0.02	0.20
Na_2O	0.10	0.12	0.17	3.60	0.05	6.50
K_2O	0.08	0.06	0.11	0.11	0.04	0.55
P_2O_5	0.07	0.02	0.23	13.20	0.25	22.45
LOI	3.26	1.22	1.98	13.83	6.23	11.37
K_r	9.83	27.29	2.07	1.54	3.23	0.40

Legend: S – Sediment Fraction; C – Concretion Fraction; ϕ - diameter; LOI – Loss on Ignition; I – 9.5mm to 2.00mm; II – 2.00mm to 0.075mm; III – 0.075mm to 0.002mm; IV - < 0.002mm; V – 0.075mm to 0.002mm; VI - < 0.002mm.

Source: Authors, 2020.

Table 3 – XRF test results of sample B2 expressed in %.

Material	**B2-S**				**B2-C**	
ϕ **(mm)**	**I (%)**	**II (%)**	**III (%)**	**IV (%)**	**V (%)**	**VI (%)**
SiO_2	69.30	70.94	43.52	39.50	66.38	37.42
Fe_2O_3	15.97	2.80	6.80	6.45	18.85	19.35
Al_2O_3	7.63	17.37	34.00	28.96	4.36	11.29
TiO_2	0.69	1.25	1.72	2.08	0.62	0.78
MnO	0.02	0.01	0.01	0.01	0.01	0.01
CaO	0.53	0.04	0.05	0.07	0.05	0.08
MgO	0.05	0.05	0.05	0.07	0.04	0.07
Na_2O	0.14	0.10	0.11	1.30	0.08	4.20
K_2O	0.10	0.06	0.05	0.05	0.07	0.18
P_2O_5	0.07	0.02	0.38	5.68	0.11	15.30
LOI	5.44	7.26	13.35	15.73	4.38	13.22
K_r	6.61	6.30	1.93	2.03	6.89	2.69

Legend: S – Sediment Fraction; C – Concretion Fraction; ϕ - diameter; LOI – Loss on Ignition; I – 9.5mm to 2.00mm; II – 2.00mm to 0.075mm; III – 0.075mm to 0.002mm; IV - < 0.002mm; V – 0.075mm to 0.002mm; VI - < 0.002mm.

Source: Authors, 2020.

Table 4 – XRF test results of sample B3 expressed in %.

Material	**B3-S**				**B3-C**	
ϕ **(mm)**	**I (%)**	**II (%)**	**III (%)**	**IV (%)**	**V (%)**	**VI (%)**
SiO_2	74.30	86.58	50.98	37.64	24.66	29.80
Fe_2O_3	9.85	0.08	2.30	2.70	49.05	30.00
Al_2O_3	10.13	7.43	31.20	27.80	13.20	22.90
TiO_2	0.62	0.42	2.08	2.08	0.78	0.62
MnO	0.01	0.01	0.01	0.01	0.02	0.01
CaO	0.08	1.21	0.04	0.07	0.02	0.14
MgO	0.04	0.04	0.05	0.07	0.05	0.07
Na_2O	0.09	0.09	0.14	0.13	0.12	1.10
K_2O	0.06	0.10	0.03	0.10	0.06	0.08
P_2O_5	0.03	0.03	0.37	0.21	0.34	1.34

LOI	4.74	3.27	12.75	16.40	11.36	13.83
K_r	7.70	19.68	2.65	2.17	0.94	1.21

Legend: S – Sediment Fraction; C – Concretion Fraction; ϕ - diameter; LOI – Loss on Ignition; I – 9.5mm to 2.00mm; II – 2.00mm to 0.075mm; III – 0.075mm to 0.002mm; IV - < 0.002mm; V – 0.075mm to 0.002mm; VI - < 0.002mm.

Source: Authors, 2020.

Table 6 summarizes the results of XRD tests. In general, the clay mineral kaolinite was present in nearly all the sediment and concretion subfractions. Quartz was also observed in all the subfractions with a diameter larger than 0.002 mm. The minerals goethite and hematite were also detected, with a greater occurrence of the former. The chemical and mineralogical aspects are discussed separately for each sample and the variation in SiO_2, Al_2O_3 and Fe_2O_3 (main oxides) in relation to the grain diameters of the sediment and concretion fractions is assessed.

Figure 6 shows the relation between the main oxide contents and average particle diameter of B1 samples. For the sediment fraction, silicon oxide content was predominant in silt and sand. The highest content of this oxide was detected for sand, which exhibits low iron and aluminum oxide contents. The highest Al_2O_3 values occur for silt, while silicon and aluminum oxides are present in equal proportions in clay. The amount of iron oxide, however, is low and varies little in relation to the diameters of this fraction. The concretion fraction, on the other hand, displays a high content of this oxidefor both clay and silt. In addition, silt has a higher SiO_2 and lower Al_2O_3 content than that of clay.

Mineralogical analyses conducted using XRD detected quartz and kaolinite in practically all the subfraction samples, in both the sediment and concretion fractions. Goethite was also identified in the silt and clay subfractions of the sediment fraction. Finally, a scanning electron microscope identified quartz particles with polished and rough textures, cemented by iron oxides, in the concretion fraction.

Table 5 – XRD results of Barreiras Formation sediments (B1, B2 and B3).

Material	ϕ (mm)	Minerals Found
B1-S	I	Quartz, Kaolinite
B1-S	II	Quartz, Kaolinite
B1-S	III	Quartz, Kaolinite, Goethite
B1-S	IV	Quartz, Kaolinite, Goethite
B1-C	V	Quartz
B1-C	VI	Quartz, Kaolinite, Goethite
B2-S	I	Quartz, Kaolinite, Goethite
B2-S	II	Quartz
B2-S	III	Quartz, Kaolinite, Goethite
B2-S	IV	Kaolinite, Goethite
B2-C	V	Quartz, Kaolinite, Goethite
B2-C	VI	Quartz, Kaolinite, Goethite, Hematite
B3-S	I	Quartz, Kaolinite
B3-S	II	Quartz, Kaolinite
B3-S	III	Quartz, Goethite
B3-S	IV	Goethite, Hematite
B3-C	V	Quartz, Goethite, Hematite
B3-C	VI	Quartz, Kaolinite, Goethite

Legend: S – Sediment Fraction; C – Concretion Fraction; ϕ - diameter; L.I. – Loss on Ignition; I – 9.5mm to 2.00mm; II – 2.00mm to 0.075mm; III – 0.075mm to 0.002mm; IV - < 0.002mm; V – 0.075mm to 0.002mm; VI - < 0.002mm.

Source: Authors, 2020.

The variation in the main oxides in relation to the average grain diameter of the B2 sample is presented in Figure 7. Silicon oxide is predominant in all the subfractions of sediment and concretion fractions, with the highest contents found in sandparticles. In the sediment fraction, aluminum oxide is more present than iron and found in a greater proportion in the siltsubfraction. In concretion, however, Fe_2O_3 content is higher than that of the sediment fraction, although SiO_2 continues to predominate. Silt exhibits lower aluminum oxide content than that of clay, but a higher silicon oxide level.

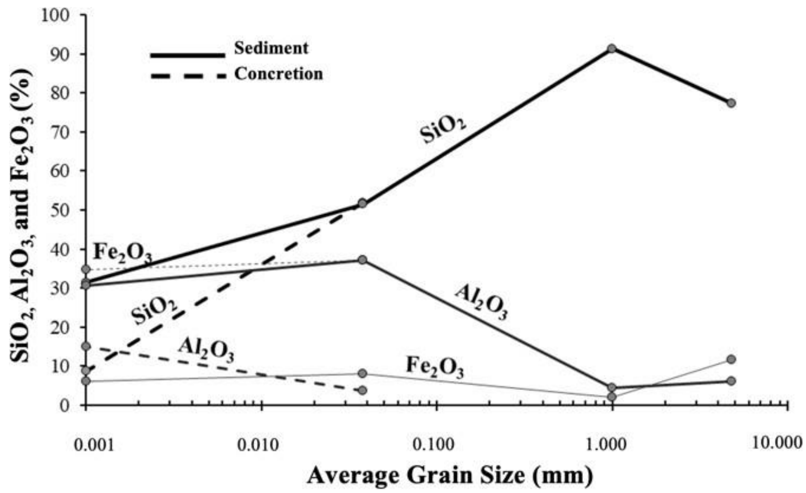


Figure 6 – Variation in SiO_2, Al_2O_3 and Fe_2O_3 content for sample B1.
Source: Authors, 2020.

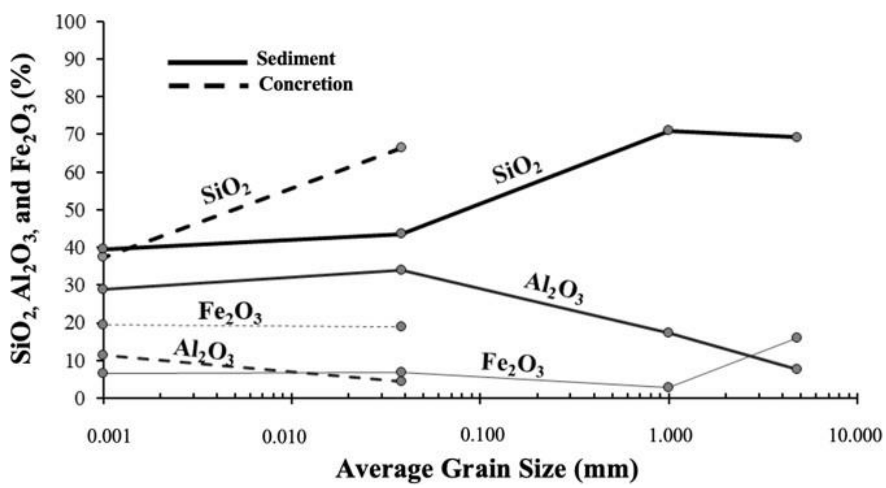


Figure 7 – Variation in SiO2, Al2O3 and Fe2O3 content for sample B2.
Source: Authors, 2020.

Mineralogical analyses of the B2 sample identified quartz, kaolinite and goethite. Hematite was also detected, but only for the clay subfraction. SEM of the concretions of this deposit showed quartz particles with a rough worn surface and grain cementation by iron oxide.

The graph of Figure 8 was obtained from the correlation between the main oxide contents and average grain diameter of sample B3. Silicon oxide is predominant in the sediment fraction for all the subfractions, and higher for the sand-size diameter, followed by aluminum and iron oxide in descending order. It is important to underscore that Fe_2O_3 is found in very small amounts in the sediment fraction. For this fraction, Al_2O_3 content is higher for silt. In relation to concretion, iron oxide content is significantly higher, with the highest amount found in the silt subfraction. Silicon and aluminum oxides are found in greater amounts for clay than silt.

The XRD diffractograms obtained for sample B3 exhibited quartz, kaolinite, goethite and hematite. Goethite was found in practically all the subfractions and hematite in claysubfractions of the sediment and concretion. SEM analysis revealed rough quartz particles cemented by iron oxides.

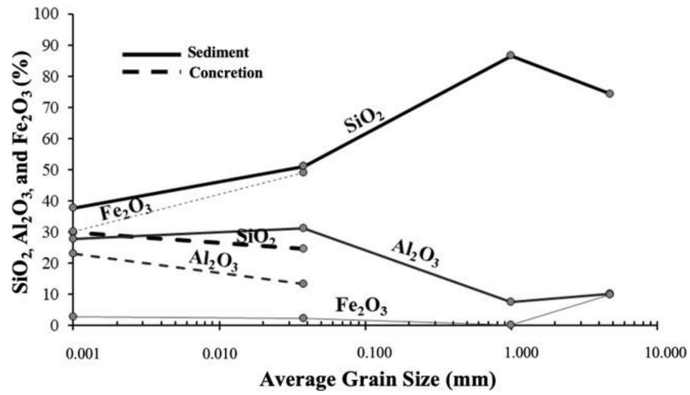


Figure 8 – Variation in SiO_2, Al_2O_3 and Fe_2O_3 content for sample B1.
Source: Authors, 2020.

4.2. Geotechnical Characterization of Samples B1, B2 and B3

The particle size curves obtained in samples B1, B2 and B3 are presented in Figure 9.The other geotechnical characterization results of the sediments are shown in Table 7.

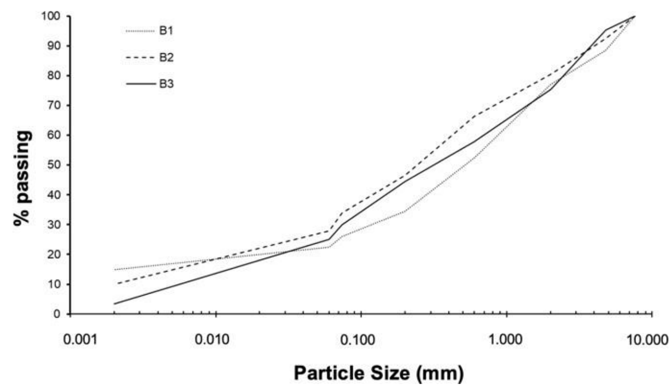


Figure 9 – Particle size curves of Barreiras Formation sediments (Samples B1, B2 and B3).
Source: Authors, 2020.

Table 6 – Geotechnical characterization of Barreiras Formation sediments (Samples B1, B2 and B3).

#	Samples		
	B1	**B2**	**B3**
γ_s (kN/m^3)	26.7	26.7	26.6
LL (%)	24.0	28.0	23.0
PL (%)	17.0	19.0	-
PI (%)	7.0	9.0	NP
Atividade	0.47	0.90	-
Classification (USCS)	SC	SC	SM

Legend: LL – Liquid Limit; PL – Plastic Limit; PI – Plasticity Index; NP – Non-Plastic
Source: Authors, 2020.

The sediments were classified as clayey sand – CS and silty sand – SM, according to the Unified Soil Classification System (USCS). Samples B1 and B2 showed low and medium plasticity, respectively. B3 sediment was classified as non-plastic. The clay fraction of sample B1, with an activity index of 0.47, is classified as inactive, while the clay in B2 is considered normal activity.

The plasticity characteristics of the materials are due to the affinity of the fine fraction of sediments with water. The fact that the materials exhibit low and medium plasticity and, consequently low clay fraction activity, is compatible with the presence of kaolinite (1:1 low activity clay mineral) and iron-rich minerals in the fine fraction (silt and clay). Low plasticity and activity were identified in other Barreiras Formation sediments in Rio Grande do Norte (Santos Jr. *et al.*, 2015), which is expected for lateritic soils. These materials exhibit strength and stress-strain behavior strongly influenced by the natural cementation formed between the particles due to the presence of iron oxides in their mineralogical composition.

5. Characterization of post-barreiras (pb) sediments

5.1. Chemical and Mineralogical Analysis of Samples PB0, PB1, PB2, PB3 and PB4

The results of XRF and XRD tests are presented in Tables 8 and 9, respectively. Sample PB0 was separated into sand and clay fractions in order to allow direct comparison with Barreiras Formation samples (divided into concretion and sediment). The others were analyzed with whole samples, without separation.

Table 7 – XRF results of Post-Barreiras sediments (Samples PB0, PB1, PB2, PB3 and PB4) expressed in %.

Material	**PB0**		**PB1**	**PB2**	**PB3**	**PB4**
ϕ (mm)	**I**	**II**	**-**			
SiO_2	71.91	31.53	61.73	56.54	46.69	54.75
Al_2O_3	10.55	34.08	30.87	36.22	37.54	37.69
Fe_2O_3	2.67	10.19	1.73	2.12	4.09	1.95
TiO_2	0.52	1.31	0.87	0.67	0.57	0.51
K_2O	-	0.33	0.14	0.12	0.10	0.13
SO_3	0.19	-	0.09	0.11	0.08	0.10
CaO	0.33	0.27	-	0.06	0.12	-
ZrO_2	0.04	0.02	0.06	0.02	0.02	0.02
Cr_2O_3	-	0.04	0.01	0.01	0.02	0.01
MnO	-	0.01	0.01	0.01	0.02	-
ZnO	0.01	0.02	-	-	-	-
CuO	0.01	0.03	-	-	-	-
P_2O_5	-	7.38	-	-	-	-
SrO	-	0.01	-	-	-	-
L.I.	13.77	14.78	4.49	4.12	7.75	4.84

Legend: ϕ - diameter; L.I. – Loss on Ignition; I – 2.00mm to 0.075mm; II < 0.002mm;

Source: Authors, 2020.

The main oxides identified in the chemical analysis of sample PB0 were silicon oxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3), for Barreiras Formation sediments (B1, B2 and B3). The chemical composition of SiO_2 was also highest in sand, followed by Al_2O_3 and Fe_2O_3. These results were used to calculate the silica/sesquioxide ratio for the clay fraction, resulting in Kr = 1.3 (less than 2), characterizing a lateritic material (Winterkorn and Fang, 1991). The amount of iron oxide present in the sand and clay fractions resembles that found for the sediment fraction of the Barreiras Formation. High iron content, associated with the formation of ferruginous concretions, was not observed for sample PB0.

Table 9 – XRD results of Post-Barreiras sediments (Samples PB0, PB1, PB2, PB3 and PB4).

Sample	**Minerals Found**
PB0 Sand Fraction	Quartz (SiO_2) and Nacrite ($Al_2Si_2O_5(OH)_4$)

PB0 Clay Fraction	Nacrite ($Al_2Si_2O_5(OH)_4$)
PB1	Quartz (SiO_2), Nacrite ($Al_2Si_2O_5(OH)_4$) and Anatase (TiO_2)
PB2	Quartz (SiO_2), Nacrite ($Al_2Si_2O_5(OH)_4$) and Anatase (TiO_2)
PB3	Quartz (SiO_2), Nacrite ($Al_2Si_2O_5(OH)_4$) and Anatase (TiO_2)
PB4	Quartz (SiO_2), Nacrite ($Al_2Si_2O_5(OH)_4$) and Anatase (TiO_2)

Source: Authors, 2020.

X-ray diffraction of sample PB0 revealed two crystalline phases: SiO_2, corresponding to quartz and $Al_2Si_2O_5(OH)_4$ to nacrite (polymorph of kaolinite). In relation to clay, only nacrite ($Al_2Si_2O_5(OH)_4$) was identified. The clay minerals with this composition are the 1:1 type, which is consistent with the low activity observed for the clay fraction in geotechnical characterization. Unlike the Barreiras Formation sediments in sample PB0, neither goethite nor hematite was identified in XRD analyses.

The subfractions of sample PB0 were analyzed by scanning electron microscopy (SEM). Figure 10 presents micrographs of sand, silt and clay particles. Figure 10a shows grains of sand at 40x magnification. In general, rough grains with low sphericity are observed, varying between angular and subangular. Figure 10b exhibits a grain of quartz with fine material incrusted in the recesses. Figures 10c and d depict silt particles surrounded by agglomerated clay particles.

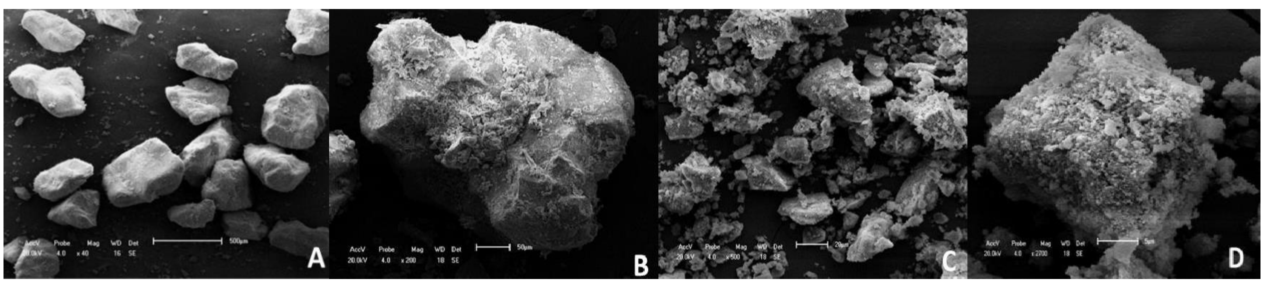


Figure 10 – SEM microphotograph,a) and b) sand (0.075 mm ≤ ϕ ≤ 2.00 mm) and c) e d) silt and clay (ϕ ≤ 0.075 mm).
Source: Authors, 2020.

In terms of samples PB1, PB2, PB3 and PB4, XRF and XRD tests found similar chemical and mineralogical compositions for the four samples. The main oxides in the four samples are silicon oxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3), as well as for sample PB0. All the Post-Barreiras samples showed a silica-sesquioxide ratio greater than or equal to 2. According to DNIT-ES guideline 098/2007, one of the requirements for a sediment to be considered lateritic is Kr < 2. As such, based on this parameter, these samples can be classified as non-lateritic.

The mineralogical analyses conducted with samples PB0, PB1, PB2, PB3 and PB4 identified quartz, nacrite and anatase. The XRD of these samples found no hematite or goethite. The similarity between the chemical and mineralogical characteristics observed for samples PB0, PB1, PB2, PB3 and PB4 suggests that they belong to the same formation.

5.2. Geotechnical Characterization of Samples PB0, PB1, PB2, PB3 and PB4

Figure 11 presents the particle size results of samples PB0, PB1, PB2, PB3 and PB4. The test results demonstrate the particle size similarity between the materials, with practically coincident curves. The results show that the Post-Barreiras sediments contain a smaller amount of gravel and higher content of fine material (which passes through a #200 sieve) than that of Barreiras Formation sediments (B1, B2, and B3).

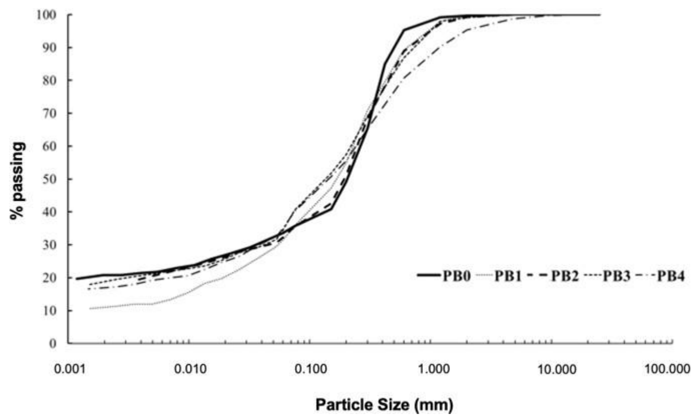


Figure 11 – Particle size curves of Post-Barreiras sediments (Samples PB0, PB1, PB2, PB3 and PB4).
Source: Authors, 2020.

Table 10 presents the results of characterization tests, where PI varied between 6 and 8%, characterizing sediments with low and medium plasticity, the clay fraction being inactive. The samples do not differ significantly from those of the Barreiras Formation in terms of plasticity and clay fraction activity, both result from the presence of 1:1 clay minerals in the chemical composition. The presence of fewer iron oxides in the Post-Barreiras sediments suggest that these soils exhibit different mechanical behavior, which should be considered when these materials are used in engineering projects. The materials were classified as clayey sand (SC) and silty-clayey sand (SC-SM). Although the collection points are located along an 85-km stretch, the materials exhibited similar characteristics in all characterization aspects.

Table 8 – Characterization of Barreiras Formation sediments (Samples PB0, PB1, PB2, PB3 and PB4).

#	Samples				
	PB0	**PB1**	**PB 2**	**PB3**	**PB4**
γ_s (kN/m^3)	26.4	26.1	26.6	25.9	26.7
LL (%)	24.0	27.0	26.0	26.0	28.0
PL (%)	15.0	20.0	20.0	20.0	20.0
PI (%)	8.0	7.0	7.0	6.0	8.0
Activity	0.40	0.65	0.37	0.29	0.48
Classification (USCS)	SC	SC-SM	SC-SM	SC-SM	SC

Legend: LL – Liquid Limit; PL – Plastic Limit; PI – Plasticity Index; NP – Non-Plastic
Source: Authors, 2020.

6. Conclusion

The present study analyzed the chemical, mineralogical and geotechnical aspects of the Barreiras and Post-Barreiras sediments in Rio Grande do Norte state, Brazil. In general, the most important chemical elements in the formation of these elements are silicon, aluminum and iron.

In relation to the distribution of the main oxides found in the Barreira Formation sediments as a function of the average grain diameter, a number of generalizations can be made: (1) the fractions with the largest diameters predominantly had the highest silicon oxide and lowest aluminum oxide content; (2) the sediment fraction is formed by silicon oxide, aluminum and iron, in descending order; (3) there is a significant difference in iron oxide content between the sediment and concretion fractions, with the accumulation of iron oxides being the main requirement for concretion formation. Mineralogical analyses revealed the occurrence of quartz, kaolinite, hematite and goethite. The concretions of samples B1, B2 and B3 demonstrated quartz particles with textures varying between polished and rough and with ferruginous cementation. In terms of geotechnical classification, the sediments were classified in the Unified Soil Classification System as SC (clayey sand) or SM (silty sand). They exhibit low to medium plasticity and clay fraction with low to medium activity, resulting from the presence of kaolinite and iron-rich minerals in the fine fraction.

Chemical analyses of the Post-Barreiras sediments (PB0, PB1, PB2, PB3 and PB4) demonstrated a lower amount of iron oxide than in the Barreiras Formation. XRD analyses identified quartz, nacrite and anatase. Hematite and goethite were not found, unlike that observed in Barreiras Formation sediments. In geotechnical terms, the sediments were classified as SC (clayey sand) or SC-SM (clayey-silty sand) with low to medium plasticity. The clay fraction exhibits low activity, attributed to the presence of 1:1 clay minerals.

Comparison between the data obtained for Barreiras and Post-Barreiras Formation sediments shows a difference in chemical composition, primarily in relation to the presence of iron oxide, responsible for the formation of natural cementation between the particles. Despite having a similar geotechnical classification, the difference in chemical and mineralogical composition may result in different mechanical behaviors between the two types of sediments. The ferruginous cementation of Barreiras Formation sediments provides cohesion to the material, which explains the presence of steep slopes, such as the cliffs along the coast of Rio Grande do Norte.

Finally, the data collected in the present study aimed to contribute to the database of Barreiras Formation soils and a better understanding of the behavior of soils on the south coast of the state, as well as the more rational use of these materials in engineering materials.

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References

ABNT. Associação Brasileira de Normas Técnicas. NBR 6459 – *Solo: Determinação do Limite de Liquidez*. Rio de Janeiro, ABNT, 2016.

ABNT. Associação Brasileira de Normas Técnicas. NBR 7180 – *Solo: Determinação do Limite de Plasticidade.* Rio de Janeiro, ABNT, 2016.

ABNT. Associação Brasileira de Normas Técnicas. NBR 6457 – *Amostras de solo - Preparação para ensaios de compactação e ensaios de caracterização.* Rio de Janeiro, ABNT, 2016.

ABNT. Associação Brasileira de Normas Técnicas. NBR 7181 – *Solo: Análise Granulométrica*. Rio de Janeiro, ABNT, 2016.

Barreto, A.M.F.; Suguio, K.; Bezerra, F.H.R.; Tatumi, S.H.; Yee, M.; Giannini, P.C.F. Geologia e Geomorfologia do Quaternário Costeiro do Estado do Rio Grande do Norte. *Revista do Instituto de Geociências - USP Geol***.**, v. 4, n. 2, p. 1–12, 2004.

BARBOSA, N.M. *Estabilidade das Falésias da Barreira do Inferno – RN.* 114p. Dissertação (Mestrado em Engenharia Civil) – Pós-Graduação em Engenharia Civil, Universidade Federal do Rio Grande do Norte, Natal, 2017.

Bezerra, F.H.R.; Mello, C. L.; Suguio, K. A Formação Barreiras: Recentes avanços e antigas questões. *Geologia USP - Serie Cientifica*, v. 6, n. 2, 2006.

DNIT. Departamento Nacional de Infraestrutura de Transportes. DNIT-ES 098: Pavimentação – base estabilizada granulometricamente com utilização de solo laterítico – Especificação de serviço. Rio de Janeiro, 2007.

Lima, C.C.U. DE; Vilas Boas, G.D.S.; Bezerra, F.H.R. Faciologia e análise tectônica preliminar da Formação Barreiras no Litoral Sul do estado da Bahia, Brasil. ***Geologia USP. Série Cientifica,*** v. 6, n. 2, p. 71–80, 2006.

LUCENA, T. S. *Efeito da variação do teor de umidade e da energia de compactação sobre as propriedades mecânicas de um solo laterítico***.** 13 p. Trabalho de Conclusão de Curso (Graduação em Engenharia Civil), Universidade Federal do Rio Grande do Norte, Natal, 2015.

PEREIRA, I.C.B.B.A. *Contribuição ao Conhecimento do Meio Físico da Região do Complexo Estuarino-Lagunar Nísia Floresta-Papeba-Guaraíras.* Dissertação (Mestrado em Engenharia Civil). Programa de Pós-graduação em Engenharia Sanitária – Universidade Federal do Rio Grande do Norte, Natal, 2004.

PEREIRA, K.L.A. *Estabilização de um solo com cimento e cinza de lodo para uso em pavimentos.* Dissertação (Mestrado em Engenharia Civil). Programa de Pós-graduação em Engenharia Civil – Universidade Federal do Rio Grande do Norte, Natal, 2012.

Rossetti, D.F.; Góes, A.M.; Bezerra, F.H.R; Valeriano, M.M.; Brito-Neves, B.B.; Ochoa, F.L. Contribution to the stratigraphy of the onshore Paraíba basin, Brazil. *Anais da Academia Brasileira de Ciencias,* v. 84, n. 2, p. 313–333, 2012.

Rossetti, D. F.; Bezerra, F. H. R.; Dominguez, J. M. L. Late oligocene-miocene transgressions along the equatorial and eastern margins of brazil. *Earth-Science Reviews*, v. 123, p. 87–112, 2013.

Santos Júnior, O. F.; Coutinho, R. Q.; Severo, R. N. F. Propriedades Geotécnicas dos Sedimentos da Formação Barreiras no Litoral do Rio Grande do Norte - Brasil. *Geotecnia,* v. 134, p. 87–108, 2015.

SEVERO, R.N.F. *Análise da estabilidade das falésias entre Tibau do Sul e Pipa – RN.* 139p. Dissertação (Mestrado em Engenharia Sanitária). Programa de Pós-graduação em Engenharia Sanitária – Universidade Federal do Rio Grande do Norte, Natal, 2005.

SEVERO, R.N.F. *Caracterização geotécnica da falésia da Ponta do Pirambu em Tibau do Sul - RN considerando a influência do comportamento dos solos nos estados indeformado e cimentado artificialmente.* Tese (Doutorado em Engenharia Civil). Programa de Pós-graduação em Engenharia Civil – Universidade Federal de Pernambuco, Recife, 2011.

SILVA, W.S. *Estudo da Dinâmica Superficial e Geotécnico das Falésias do Município de Tibau do Sul – Litoral Oriental do RN.* 132p. Dissertação (Mestrado em Engenharia Sanitária). Programa de Pós-graduação em Engenharia Sanitária – Universidade Federal do Rio Grande do Norte, Natal, 2003.

SOUSA, R. DE A. *Resistência e Compressibilidade de Solos da Formação Barreiras da Região de Natal/RN.* 87p. Dissertação (Mestrado em Engenharia Civil). Programa de Pós-graduação em Engenharia Civil – Universidade Federal do Rio Grande do Norte, Natal, 2018.

SOUZA JR., C. *Análise da Estabilidade de Falésias na Zona Costeira de Baía Formosa/RN.* Dissertação (Mestrado em Engenharia Civil). Programa de Pós-graduação em Engenharia Civil – Universidade Federal do Rio Grande do Norte, Natal, 2013.

SOUZA, T. A. C. de. *Efeito da variação da energia de compactação sobre as propriedades mecânicas de um solo laterítico.* 18p. Trabalho de Conclusão de Curso (Graduação em Engenharia Civil) - Universidade Federal do Rio Grande do Norte, Natal, 2016.

TAQUEZ, D. E. D. *Susceptibilidade à ocorrência de movimentos de massa e avaliação da estabilidade de falésias sob condição não saturada: Estudo de caso no Centro de Lançamentos da Barreira do Inferno – Brasil*. Dissertação (Mestrado em Engenharia Civil). Programa de Pós-graduação em Engenharia Civil – Universidade Federal do Rio Grande do Norte, Natal, 2017.

Vilas Bôas, G. S.; Sampaio, F. J.; Pereira, A. M. S. The Barreiras Group in the northeastern coast of the State of Bahia, Brazil: Depositional mechanisms and processes. *Anais da Academia Brasileira de Ciencias*, v. 73, n. 3, p. 416–427, 2001.

WINTERKORN, H.F.; FANG, H.Y. Soil Technology and Engineering Properties of Soils. In: FANG, H.Y. *Foundation Engineering Handbook.* Chapman & Hall, New York, 1991. p. 88-143.