Soil characterisation used in ceramic industries and an analysis of its feasibility in ecological bricks

Caracterização de solos utilizados em indústrias cerâmicas e análise da visibilidade de uso em tijolos ecológicos

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Abstract

n this study, we aimed to characterise soils of three sedimentary deposits used as raw material sources for ceramic industries located in the Brazilian semi-arid region. The samples were collected in deposits located in the cities of Barra de São Miguel, Juazeirinho and Santa Cecília. They were named, prepared and submitted to tests to determine the mineralogical, chemical, physical and thermal characteristics using the XRD, EDX, Casagrande, granulometric analysis, TG and DTA. The soils presented compositions containing quartz, alumina and hematite, predominantly with about 90% of the total, with well-defined peaks in the diffractograms confirming the statement, and averages of plasticity and granulometric distribution that fit them as silt-clay. When heated to 1000 °C, fire losses were 17%, 16% and 29% for samples from Barra de São Miguel, Juazeirinho and Santa Cecília, respectively, which is due to the dehydration and burning of organic matter. Thus, they have met the requirements of the Brazilian standards that deal to produce sealing (with sintering) and soil-cement bricks (by pressing). The studied deposits offer satisfactory raw material for the ceramics industry and can provide soil for soil-cement brick (ecological bricks) production.

Keywords: Soil evaluation. Mineralogy. Semi-arid soils. Particle size analysis.

Resumo

Neste estudo, objetivamos caracterizar solos de três depósitos sedimentares utilizados como fontes de matéria-prima para indústrias de cerâmica localizadas no semiárido brasileiro. As amostras foram coletadas em depósitos localizados nos municípios de Barra de São Miguel, Juazeirinho e Santa Cecília, nomeadas, preparadas e submetidas a testes para determinação das características mineralógicas, químicas, físicas e térmicas pelas técnicas XRD, EDX, Casagrande, análise granulométrica, TG e DTA. As composições dos solos continham quartzo, alumina e hematita, predominantemente, cerca de 90% do total, com picos bem definidos nos difratogramas que confirmam a afirmação, e apresentaram plasticidade e distribuição médias de grãos que os encaixam como silto-argilosos. Quando aquecidas até 1000 °C, as perdas ao fogo foram de 17%, 16% e 29% para as amostras de Barra de São Miguel, Juazeirinho e Santa Cecília, percentual devido à desidratação e queima de matéria orgânica. Assim, os solos estudados atenderam aos requisitos das normas brasileiras que tratam da produção de tijolos de vedação (com sinterização) e de solo-cimento (por prensagem). Os depósitos estudados oferecem matéria-prima satisfatória para a indústria cerâmica e podem fornecer solo para a produção de tijolos solo-cimento (tijolos ecológicos).

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Introduction

Soils are defined as a material layer of variable consistency found above the lithosphere and below the atmosphere containing minerals, organic matter, water, water solutions with other components and air representing, respectively, the solid, liquid and gaseous phases, resulting from physical, chemical and biological processes that decompose the matrix rocks, with variations according to the environmental conditions of each region (KUČERÍK; ČTVRTNÍČKOVÁ; SIWERT, 2013; MILLIOLI *et al.*, 2009; AÑÓN *et al.*, 2007; REGUEIRA *et al.*, 2006). It is a natural resource considered as renewable, but which undergoes constant changes in its characteristics due to inclement weather and, mainly, through direct exploration or the activities that increase erodibility, alter salinity and insert contaminating substances, among others (MARSAN *et al.*, 2019; VAEZI; HASANZADEH; CERDÀ, 2016; WEISSERT; SALMOND; SCHEWENDENMANN, 2016; XIA *et al.*, 2016; KUČERÍK; ČTVRTNÍČKOVÁ; SIWERT, 2013).

Each area of science studies and works the soil in a particular way: biology sees the soil by the conditions that allow the diversity of life, chemistry as a set of organic and inorganic components, engineering on the conditions of resistance and characteristics that allow the extraction for creating construction pieces or utensils of the most diverse uses, agronomy use for cultivation or pastures, among others, also having areas that, from knowledge about soil characteristics defined by others, for example medicinal uses of components of the soil characterized by chemistry (HARTEMINK, 2016). According to Hartemink (2016), each subdiscipline has its own definition for soils, focusing on its scientific line, reinforcing that the definition may be simple or complex, depending on who proposes it.

In this perspective, Bernatek-Jakiel, Kacprzak and Stolarczyk (2016) studied soil characteristics in piping or "piping" activities and their behaviour on the formation of what are defined as "underground pipes" and observed that biological activities, composition, porosity and density have a direct influence on the results. Byre *et al.* (2016) conducted a study on soil respiration, carbon stock and stocking capacity and the direct linkage of these factors with organic matter decomposition, soil moisture and temperature, showing low stability for large areas, specifically in the southeastern United States. Lessovaia *et al.* (2016) researched the development of soil in rocks defined as "basic" and "ultrabasic" based on their porosity and mineralogy and showing as a final result that a soil originates more clay minerals with the accumulation of silicate and with less porosity.

In general, many researchers have published results about various soils around the world. By observing some of these, it can be inferred that there is a wide range of fields interested in soils:

(a) evaluation of microbial activities in agricultural volcanic soils contaminated with metals (PARELHO *et al.*, 2016);

(b) analysis of the mobility of land vehicles based on a set of time series on soil moisture, captured by WindSat and LIS satellites and by local sensors (STEVENS; MCKINLEY; VAHEDIFARD, 2016);

(c) dynamic characteristics of soil properties in Robinia pseudoacacia vegetation and coastal eco-restoration (MAO *et al.*, 2016);

(d) effect of soil properties on the absorption of pharmaceutical products by worms (CARTER; RYAN; BOXALL, 2016);

(e) the effect of two pesticides on the biology of a soil subjected to severe drought (FRANCO-ANDREU *et al.*, 2016);

(f) development of food webs in the soil of microbes and nematodes under different agricultural practices during the initial phase of pedogenesis of a Mollisols (LI *et al.*, 2016);

(g) soil temperature dynamics on a basin scale (KUNKEL; WELLS; HANCOCK, 2016);

(h) red ceramics of dangerous sludge composites with smelting sand, glass residues and acid neutralizing salts (MYMRIN *et al.*, 2016);

(i) introduction of corn fibres as reinforcement, aiming at improvements of mechanical properties of cemented soil (TRAN; SATOMI; TAKAHASHI, 2018);

(j) stabilisation of cotton soils, using limestone, volcanic ash and mixtures of these elements, observing physical-mechanical properties, ideal proportions of stabiliser mixtures and changes in related mineralogical phases (CHENG *et al.*, 2018);

(k) investigation of the effect of performance variability of cement treated soil slabs (PAN et al., 2018); and

(1) study of anthropic soils in different physical, chemical and climatic conditions to characterise physicochemical properties, focusing on the quality and quantity of organic matter (KERN *et al.*, 2019).

Clays are low-grain minerals with grains of less than 2 μ m due to the decomposition of sedimentary rocks composed of clay minerals, mainly aluminum and iron silicates, organic materials, soluble salts, oxides and other minerals (SANTOS, 1992, 2009; GUGGENHEIM; MARTIN, 1995; VELDE; MEUNIER, 2008). They are divided fundamentally into three large groups, listed by origin, physicochemical characteristics and use in the production of materials: red, white and refractory (MENEZES; NEVES; FERREIRA, 2001). There is, therefore, a great heterogeneity among the clays, and it can derive several subclassifications of this material (MACEDO *et al.*, 2008). The red clay used to produce bricks and tiles, floor tiles and tiles, exemplifying, respectively, groups of red ceramic materials with porous mass and semi-glazed mass, is called this because after firing at temperature ranges between 800 and 1250 °C, a red color (Figure 1) is obtained justified by the presence of high levels of iron oxide III (Fe₂O₃) (SANTOS, 1992; CABRAL JUNIOR *et al.*, 2005; VIEIRA; PINHEIRO, 2013; ALONSO-SARTUDE *et al.*, 2012; COSTA, 2014; HAJJAJI *et al.*, 2016). According to Santos (2009), this is a material that, when wet, presents considerable plasticity that allows processing or conformation.

Clay, as a raw material for ceramics, is used to produce light aggregates and has the advantages of offering low density and high strength, which is beneficial for structural parts, even when the parts are sintered at lower temperatures (AYATI *et al.*, 2018).

The red ceramic industry represents 40% of the Brazilian ceramic segment and consumes about 70,000,000 ton year-1 of raw material, most of which are small industrial plants (MACEDO *et al.*, 2008). The Southeastern and Southern regions of Brazil are the most developed in this activity, but other regions, notably the Northeast, have developed considerably (ASSOCIAÇÃO..., 2015; FEDERAÇÃO...; FUNDAÇÃO..., 2013). There is no control over the extraction of natural resources for use as a raw material or input in the ceramic industry, which is not exclusive to this industrial line, as well as the environmental impacts caused after withdrawal, especially during production, resulting in the degradation of the condition soil, water and air (ACCHAR; SILVA; SEGADÃES, 2013; SALEIRO, 2010; PALUDO; SANT'ANNA, 2003). Research on ceramic materials is ongoing, seeking the most diverse technological innovations, some of which are concerned with product improvements linked to the reduction of negative environmental impacts (LIU *et al.*, 2016).

Based on the importance previously described for the ceramic market and the need to gain more in-depth knowledge about materials, the objective of this work was to perform and present a physical and mineralogical characterisation of soils from three sedimentary deposits used as raw material sources for ceramic industries in the state of Paraíba, Brazil.



Figure 1 - Red clay - characteristic coloring

Materials and methods

Soil sample collections

Soil samples were collected manually using specific tools such as hoes, shovels and plastic bags for storage. The names of the samples are shown in Table 1, including the coordinates of the collection sites according to the portable GPS of the Garmim[®] brand, model eTrex 30x.

Particle size analysis

The determination of the samples grain sizes was done using a Cilas 1064 particle size analyzer. The analyses were performed in a liquid medium (distilled water), with an analysis range of 0.04 to 500 μ m. The tests were carried out to determine the characteristic particle diameters of the samples: mean diameter (dm) and diameters 10% (d₁₀), 50% (d₅₀) and 90% (d₉₀).

Atterberg boundaries

The plasticity characteristics – liquidity limit (LL), plasticity limit (PL) and plasticity index (PI) – of the soil samples were determined using the Casagrande method, which is shown in detail in ABNT (2016), NBR 7180/16, considering the results of compliance with the values specified in ABNT (2012a), NBR 10833, when dealing with ecological bricks.

X-Ray Diffraction (XRD)

The X-ray diffraction technique was used to determine the crystalline phases of the samples of the studied soils.

The diffractograms of the samples were obtained using Shimadzu XDR-6000 equipment, having as analytical conditions the scanning "2theta-theta" or " 2θ - θ " of 5° to 30°, goniometer speed of 2° min-1, step 0.02°, by the fixed time method, with CuK α radiation – 40kV and 30mA – and in an aluminum sample port.

Energy Dispersive X-ray fluorescence spectroscopy (EDX)

The energy dispersive X-ray fluorescence spectroscopy technique was used to determine the chemical composition of soil samples.

The equipment used was the Shimadzu EDX-720 model, which has a rhodium tube (Rh) and readings were made at high voltage (50 kV) and low voltage (15 kV). It detected elements between (Ti-U) and sodium and scandium (Na-Sc) and identified the peaks of energy released by the elements.

The percentage of the oxides SiO₂, Al₂O₃, Fe₂O₃, K₂O, MgO, CaO, TiO₂, BaO, SO₃, MnO, P₂O₅, SrO, Rb₂O and C were determined by the equipment-specific software.

Thermal analysis

The thermogravimetry (TG) and differential thermal analysis (DTA) techniques were used to determine the characteristics of the masses when submitted to heating, such as fire loss, humidity and organic matter content.

The equipment used was the Shimadzu DTG-60 Simultaneous model (simultaneously performing TG and DTA), with heating at 25 to 1000 °C at a rate of 10 °C min-1 in a dynamic N2 atmosphere.

Name	Description	Coordinates	Altitude (m)
SBSM	Small dam bed on Angicos Site, Barra de São Miguel	07°46'02.45"S; 36°21'54.6"W	501
SJUA	Dam bed, city of Juazeirinho	07°04'48.67"S; 36°35'29.46"W	544
SSCE	Mining area at Zé de Moura Site, city of Santa Cecília	07°44'57.05"S; 35°53'34.53"W	560

Table 1 - Detailed identification of samples

The percentages of mass losses and peak event temperatures were determined in the Shimadzu TA-60 software, version 2.20.

Results and discussion

Mineralogical characteristics of soil samples

The results of the tests by EDX – chemical composition – are shown in Table 2 for the soil samples from Barra de São Miguel, Juazeirinho and Santa Cecília, with the corresponding mass percentages of each of the oxides identified and calculated by the same equipment.

It can be observed that the compositions of the tested soils have the predominance of silica (SiO₂ or silicon dioxide), alumina (Al₂O₃ or aluminum oxide) and hematite (Fe₂O₃ or iron oxide) in common, which totalize in percentages approximately 89.5%, 89% and 86%, respectively, for samples SBSM, SJUA and SSCE, which are considered as characteristic soils of raw material sources of the red ceramic industry. The presence of potassium oxide (K₂O), calcium oxide (CaO), magnesium oxide (MgO) and titanium dioxide (TiO₂) in concentrations between 1 and 5% were also identified, while other oxides such as sulfur (SO₃ or sulfur trioxide), manganese oxide II (MnO), strontium oxide (SrO), rubidium oxide (Rb₂O) and carbon (C), appeared in very low concentrations.

Moreover, for SBSM, barium oxide (BaO) and phosphorus pentoxide (P_2O_5), for soil B, P_2O_5 , zirconium (ZrO₂ or zirconium dioxide), vanadium anhydride (V_2O_5) (III) and Y_2O_3 (yttrium (III) oxide), and for soil C, ZrO₂, V_2O_5 , Cr₂O₃, zinc oxide (ZnO) and Y_2O_3 .

The results of the XRD tests are shown in Figure 2, Figure 3 and Figure 4, respectively, for the soil samples from Barra de São Miguel, Juazeirinho and Santa Cecília, indicating characteristic peaks of elements present in the sample, and shown in Table 3.

Table 2 - Ch	emical com	position of	soil samples

Ovides	Mass (%)		
Oxides	SBSM	SJUA	SSCE
SiO ₂	56.994	51.430	48.000
Al_2O_3	23.133	24.334	26.000
Fe_2O_3	9.358	13.134	12.000
K_2O	2.985	4.020	2.000
CaO	2.950	2.037	5.000
MgO	2.281	2.553	5.000
TiO ₂	1.124	1.745	1.000
Other oxides*	1.175	0.747	1.000

Note: *other oxides: MnO, SO₃, P₂O₅, ZrO₂, BaO, V₂O₅, SrO, Cr₂O₃, Rb₂O, ZnO and Y₂O₃ < 0.2% wt (each).





Note: M: mica; H: hematite; K: kaolinite; Q: quartz; and F: feldspar.

Figure 3 - Diffractogram of the SJUA



Note: M: mica; H: hematite; K: kaolinite; Q: quartz; and F: feldspar.

Figure 4 - Diffractogram of the SSCE



Note: M: mica; I: illite; K: kaolinite; H: hematite; Q: quartz; and F: feldspar.

Analysing the diffractogram of the SBSM, the crystalline phases of quartz (SiO₂) or "Q", with peaks at $2\theta = 20.92^{\circ}$ and $2\theta = 26.67^{\circ}$ were identified, the latter being the higher relative intensity peak, confirming the highest percentage in the chemical composition of this soil; feldspars or "F", with peaks in $2\theta = 22.09^{\circ}$, $2\theta = 23.72^{\circ}$, $2\theta = 25.65^{\circ}$, $2\theta 27.42^{\circ}$, $2\theta = 27.93^{\circ}$ and $2\theta = 28.06^{\circ}$; kaolinite (Si₂Al₂O₅(OH)₄) or "K", with peaks at $2\theta = 13.88^{\circ}$ and $2\theta = 24.40^{\circ}$; mica or "M", with peaks in $2\theta = 8.89^{\circ}$, $2\theta = 17.88^{\circ}$ and $2\theta = 19.85^{\circ}$; and silica-alumina hydroxide (NaCa₂(Fe²⁺4Al)Si₆Al₂O₂(OH)₂) or "H" with peaks at $2\theta = 10.62^{\circ}$ and $2\theta = 28.47^{\circ}$.

After analysing the SJUA sample (Figure 3), the identified crystalline phases were: quartz with peaks at $2\theta = 21.03^{\circ}$ and $2\theta = 26.81^{\circ}$, whereby the latter was the higher relative intensity peak, also confirming the higher percentage in the chemical composition of this soil; feldspars, with peaks at $2\theta = 27.63^{\circ}$ and $2\theta = 27.99^{\circ}$; kaolinite, with peaks at $2\theta = 13.82^{\circ}$, $2\theta = 13.88^{\circ}$, $2\theta = 13.88^{\circ}$ and $2\theta = 24.40^{\circ}$; mica, with peaks at $2\theta = 8.89^{\circ}$, $2\theta = 17.88^{\circ}$ and $2\theta = 19.85^{\circ}$; and silica-alumina hydroxide with peaks at $2\theta = 10.62^{\circ}$ and $2\theta = 28.47^{\circ}$.

In the diffractogram of the SSCE (Figure 4), the crystalline phases of quartz, with peaks at $2\theta = 20.92^{\circ}$ and $2\theta = 26.67^{\circ}$ were identified, the latter being the peak of greatest relative intensity, confirming the highest percentage in the chemical composition of this soil; feldspars, with peaks in $2\theta = 22.09^{\circ}$, $2\theta = 23.72^{\circ}$, $2\theta = 25.65^{\circ}$, $2\theta 27.42^{\circ}$, $2\theta = 27.93^{\circ}$ and $2\theta = 28.06^{\circ}$; kaolinite, with peaks at $2\theta = 13.88^{\circ}$ and $2\theta = 24.40^{\circ}$; mica, with peaks at $2\theta = 8.89^{\circ}$ (this coinciding with the illite), $2\theta = 17.88^{\circ}$ and $2\theta = 19.85^{\circ}$; and silica-alumina hydroxide with peaks at $2\theta = 10.62^{\circ}$ and $2\theta = 28.47^{\circ}$.

According to Carreiro *et al.* (2016), Santos (2016), Luz *et al.* (2005) and Santos (1992), the kaolinite, a phase found in all studied samples, is responsible for providing plasticity of the mass and mechanical resistance to the shaped bodies in red ceramic masses. The quartz phase in the ceramic mass, in turn, offers,

among others, the characteristics of chemical stability and hardness, probably due to the crystal ordering, to the pieces produced (MASON; THOMPSON, 2010). Thus, the soil samples from Barra de São Miguel, Juazeirinho and Santa Cecília present essential characteristics to produce ceramic pieces: plasticity and stability. Specifically observing the work with red ceramics, the iron oxide III contents confirms these characteristics for the material.

Physical characteristics of samples

The results of the Casagrande tests for the Atterberg limits - liquidity limit (LL) and plasticity limit (PL) – are shown in detail in Appendix D, and summarized in Table 3, including the results of the plasticity index of samples SBSM, SJUA and SSCE.

According to the soil classification ranges from the plasticity index (which reports how clayey a soil is), presented by Caputo (1988), the soil samples studied are therefore all framed in the "medium plastic" range, as they show PI values between 7% and 15%.

Based on the classification given by Doat *et al.* (1979), the soils are classified as "silty soil", since they have PI between 5% and 25% and LL between 20% and 50%.

In the Unified Soil Classification System (USCS), these soils are classified as inorganic clays of medium plasticity, code "CL", because LL values are between 30 and 50%.

Another classification is given by the Highway Research Board (HRB), which shows the results obtained for the soil samples of the deposits from Barra de São Miguel and Santa Cecilia classifying them as "A-6" (clay soils). The results obtained from the Juazeirinho deposit soil sample only fit this in the case of rounding the results as all the classifications provide LL = 40% as the maximum value, 41% as the minimum value, PI = 10% as the maximum value and 11% as the minimum value. Therefore, with approximations, all soils are classified as clayey, defining their physical similarity.

It is also worth mentioning plasticity data, based on NBR 8491, in which all the soils worked in this research (after preparation) meet the requirements for soil-cement brick production in manual or hydraulic pressing because they have $LL \le 45\%$ and $PI \le 18\%$ (ABNT, 2012b).

Regarding granulometry, the results of the granulometric analysis with the characteristic curve are presented in Figure 5 for the Barra de São Miguel sample, Figure 6 for Juazeirinho and in Figure 7 for Santa Cecilia. Moreover, Table 4 shows the characteristic particle diameters of these soils.

Sample	Liquidity limit (LL)	Plasticity limit (PL)	Plasticity index (PI)
SBSM	$36.17 \pm 4.25\%$	$25.00 \pm 8.33\%$	$11.17 \pm 8.49\%$
SJUA	$40.01 \pm 1.86\%$	$29.19 \pm 4.84\%$	$10.82 \pm 3.93\%$
SSCE	$32.45 \pm 7.88\%$	$19.33 \pm 8.94\%$	$11.43 \pm 4.48\%$

Table 3 - Atterberg limits of the soil samples studied

Figure 5 - Diffractogram of the SBSM



Figure 6 - Diffractogram of the SJUA



Figure 7 - Diffractogram of the SSCE



Table 4 - Characteristic particle diameters

Sample	d ₁₀ (µm)	d50 (µm)	d ₉₀ (µm)	$\mathbf{d}_{\mathbf{m}}\left(\boldsymbol{\mu}\mathbf{m}\right)$
SBSM	0.97	9.80	31.67	13.57
SJUA	1.12	8.10	26.23	11.17
SSCE	1.15	10.52	27.61	12.56

Since the fraction with grains up to 2 μ m is defined as clay, this corresponds to 20.23% of the total volume of the soil sample SBSM, 18.73% of the sample of SJUA and 17.15% of the SSCE, while in the same order, the fraction with particle sizes between 2 and 60 μ m in diameter, defined as silt, characterizes 79.77%, 81.27% and 82.85% (SANTOS, 1992; ABNT, 2012a).

The results of the particle size analysis corroborated with what the Atterberg limits indicated: silt-clay soils.

Thermal characteristics of soil samples

The soil samples were treated and submitted to TG and DTA thermal tests. TG, DTG and DTA curves of the SBSM, SJUA and SSCE samples are shown, respectively, in Figure 8, Figure 9 and Figure 10.

By analysing the behaviour of soil samples submitted to heating, it can be observed in the TG curves that there were three defined mass loss events, seen more clearly observing the DTG curves. In an analytical way, the first event, the dehydration of the material or the release of water or its solutions commonly present in soils, occurs up to a temperature of 200 °C, showing losses of 8.425%, 6.822% and 4.273%, respectively, for the SBSM, SJUA and SSCE samples (SILVA *et al.*, 2016). The second event, initiated at 200 °C,

resulted in mass losses of 7.988% up to 679 °C for SBSM, 8.725% up to 765 °C for SJUA and 24.376% up to 880 °C for SSCE. Until the end of heating, the masses of the samples suffered reductions of 0.663% in SBSM, 0.710% in SJUA and 0.262% in SSCE. The DTG and DTA curves and peaks confirm the events occurring with the heated masses.



Figure 8 - TG, DTG and DTA curves of the SBSM sample

Figure 9 - TG, DTG and DTA curves of the SJUA sample



Figure 10 - TG, DTG and DTA curves of the SSCE sample



Regarding the aforementioned numbers, it can be stated that the loss to fire totalled 17.076% for SBSM, 16.257% for SJUA and 28.911% for SSCE, in which this mass was associated with free water and volatilized solution and burning of organic matter, showing that the total and the mass losses in the second temperature range analysed were more accentuated for the SSCE sample, which presented, therefore, a higher content of organic matter (GONÇALVES *et al.*, 2017; CARTAXO *et al.*, 2016; DIAS; LIMA, 2004; ABNT, 1996). According to Gonçalves *et al.* (2017), the peak observed in the DTA curve in the 700 and 800 °C range may be associated with the onset of clay sintering and changes in the crystal structure.

Conclusions

The compositions of the soil samples from Barra de São Miguel, Juazeirinho and Santa Cecilia were similar, with a predominance of silica, alumina and hematite, comprising about 90% of each plasticity, as medium plastics, and the granulometry, in the range of "silt-clay", with variations in the thermal behaviour. They meet the standard requirements of soils that produce pressed bricks. The results obtained showed that the raw materials meet the technical specifications required for use in soil-cement bricks (a type of ecological brick) and sealing bricks (the most used in construction). Specifically, for bricks that are produced in a conventional way (with burning), thermal analysis showed masses with behaviour common to those presented by raw materials from the red ceramic industry.

Therefore, the deposits where the samples were collected offer satisfactory raw material for the ceramics industry.

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