The influence of compositional variability of dimension stone residues on the properties of rustic porous ceramic tiles

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Abstract

This study analyzed how the compositional variability of dimension stone residues influences the characteristics of rustic porous ceramic tiles. The specimens were prepared by adding 20 wt% of dimension stone residue to the clay and sintered at 1000 °C for 1 h. The testing assays performed were physical properties and structural analysis through scanning electron microscopy. The results indicate that water absorption was lower (~9% to 14%) in samples containing silicate residues compared to carbonate residues (~16%). Apparent porosity of ~21% to 25% and shrinkage of 0.5% to 1.6% were measured in silicate samples while higher apparent porosity of ~30% and expansion of -0.04% were found in carbonate samples. Flexural strength and modulus of rupture were similar, ~16 MPa and ~330 N, respectively, for both sample sets. The samples containing silicate residues whose SiO₂ content was between 48% and 58% and the sum of alkaline and alkaline earth oxides was less than 18% were suitable for manufacturing rustic porous ceramic tile, while residues with high SiO₂ or carbonate contents impaired the properties of the ceramic bodies. Therefore, it is recommended that the dimension stone residues intended for use in ceramic tiles be previously separated according to chemical and mineralogical characteristics.

Keywords: processing sludge, dimension stone residue, rustic porous ceramic tile, terracotta, technological properties.

INTRODUCTION

Brazil stands out in the dimension stone sector worldwide. In 2020, the Brazilian export of natural rock materials for ornamentation and coating reached US\$ 987.40 million and 2.16 million tons [1]. The State of Espírito Santo (ES), in Southeastern Brazil, is the main producer in this country. Between January and July 2021, ES was responsible for 76.09% of the dimension stone mass exported by Brazil (in financial terms, the State portion corresponded to 82.12% of the total). Considering only the exports from ES in this period, a little more than 400 thousand tons of granite rocks, marbles, and similar were exported as non-processed blocks while plates and other parts reached 628.4 thousand tons. Among the processed rocks, granite composition products are exported in much higher quantities than marble products [2]. Most of the exported materials undergo beneficiation processes before leaving the country (about 55% by volume) [1], such as cutting blocks into sheets. The quantity of residue generated during the sawing of rock blocks to compose semi-finished sheets is remarkable [3, 4]. This residue consists of very thin mud containing rock dust, steel

*ana.prado@ufca.edu.br https://orcid.org/0000-0003-4337-9404 shot, lime, and water when conventional looms are used in the process whereas the residue contains rock dust and water when diamond wire looms are used [5]. The dimension stone residue from silicate rocks generally consists of quartz, feldspars, calcite, and micaceous minerals [6] in different quantities while the residue from carbonate rocks contains calcite and dolomite. Using byproducts such as mining and ore processing residues can minimize the environmental impacts and reduce costs when manufacturing red ceramic products. The residues can be added to the product when their composition is similar to the traditional raw material used and/or when their properties are similar or better than the products existing on the market.

To this end, many works investigated including dimension stone residues in red ceramics products [6-13]. The term 'red ceramic' includes several products with clays that contain iron oxides and hydroxides, responsible for the reddish color of the pieces after firing. Among the products are roof tiles, blocks, bricks, and red ceramic tiles used on floors. Despite being extremely variable, the mineralogical composition of Brazilian clays contains non-plastic minerals that are also part of the main types of rocks used for ornamental purposes. Therefore, dimension stone residues present a great potential to be used in red ceramic products [7]. But, while some studies show that mixtures containing residue from the cutting of the dimension stone using diamond wire looms can be used to manufacture roofing tiles and structural red ceramic blocks [6], other works [8] report that mechanical resistance and water absorption decrease with the increasing percentage of dimension stone residue added to the ceramic mixture. From an economic point of view, red ceramic tile for floors (also known as 'porous rustic tile', 'terracotta', or 'baked earth') has greater added value than other red ceramic products [9]. Additionally, the manufacturing process of rustic porous ceramic coating is simpler than those employed in other ceramic types. The first product is shaped through extrusion, not enameled, and sintered at ~1000 °C, and, generally, the rustic coating has high water absorption as well as varying color and size. Although the Brazilian Association of Technical Standards (ABNT) does not establish a specific requirement for this kind of product, and some rustic porous coatings are made by pressing, their properties can be correlated with the class AIII parameters detailed in the ABNT ISO 13006/2020 standard [14].

The authors [10] that investigated the incorporation of residues from gneisses for producing rustic porous tiles recommend new tests at a sintering temperature of ~1000 °C. Another research [11] added residues from granitic rocks and kaolin to the ceramic mixture and reported that the specimens met the ABNT standard requirements for semi-porous ceramics at the sintering temperature of 1150 °C. The use of carbonate residues was also tested with different types of clays and various sintering temperatures, confirming the possibility of producing porous ceramic coatings when using kaolinitic clay and carbonate residues [12]. In the literature, a review article [7] analyzing the use of residues in red ceramic reports that, despite generating products that comply with the regulations and contribute to reducing environmental impacts, knowledge gaps still exist in the incorporation field during the manufacturing process. Nonetheless, major research investigated mixed residues, composed of mixing wastes that originated from different types of materials. In Cachoeiro

de Itapemirim, a city in the South of Espírito Santo-Brazil, the only two works that studied the residues segregated according to rock type report that the compositional variability of residues is substantial, therefore requiring to select residues according to the lithological types to obtain more valuable raw materials [5, 13]. Therefore, the present work aims to investigate how the compositional differences of the rocks processed in the Cachoeiro de Itapemirim region (ES) influence the properties of rustic porous ceramic tiles.

MATERIALS AND METHODS

The raw material used in this research was the conventional clay used to manufacture bricks in the red ceramic industry and residues from the sawing of dimension stone blocks in companies in Cachoeiro de Itapemirim (ES, Brazil). The characteristics of the clay used here have already been published [12]. The wide variety of chemical and mineralogical compositions observed in the field of dimension stones consequently generates different residue types [5]. Therefore, we characterized previously several residues from the sawing of dimension stone blocks and selected one residue per group, according to the chemical characteristics (Table I) determined by an X-ray fluorescence spectrometer (XRF, PW-2400, Philips). The mineralogical composition of the samples was determined by X-ray diffraction (XRD) using a diffractometer (X'Pert, Panalytical) that allowed measurements in the 12 to 310 K range, equipped with a copper anode (K α 1.5418 Å); the range of 2θ was from 4° to 70° , with a 0.05° step.

Silicate and carbonate rock residues were collected on conventional and diamond wire looms, respectively. Grinding was not necessary because these particles were very fine, thus, they were just dried and crushed in a mortar. The clay was wet ground for 24 h in a ball mill. Then, the clay paste obtained was first left to air dry, followed by oven drying for 24 h, and sieved through an ASTM #200

plates.																				
Table I	- C	ommer	cial	names	of th	e dim	nension	stones	and	charact	teristics	of t	he sawd	ust re	esidues	resulting	from	the c	utting	, of

Commercial name of the rock	Characteristics of the sawdust residue						
Crema Bordeaux	Silicate composition with high silica content and low LOI						
Verde Peacock	Silicate composition with intermediate SiO ₂ level, relatively high percentages of Al_2O_3 , Na_2O+K_2O , and low LOI						
Ocre Itabira	Silicate composition with intermediate SiO ₂ level, relatively high percentages of Al ₂ O ₃ , MgO+CaO, Na ₂ O+K ₂ O, and low LOI						
Nero Marinace	Silicate composition with intermediate SiO ₂ level, a high percentage of MgO+CaO, and high LOI						
Marrom Bahia	Silicate composition with low SiO ₂ content, relatively high percentages of Al_2O_3 , Na_2O+K_2O , and low LOI						
Preto Aracruz	Silicate composition with low SiO ₂ level, a high percentage of MgO+CaO, and an atypically high percentage of P_2O_5						
Mármore Aquarela	Carbonate composition with very high levels of CaO, MgO, and high LOI						

LOI: loss on ignition.

sieve (less than 75 µm) to de-agglomerate and improve conditions for the compaction process. Ten specimens were produced for each residue type by mixing it with 80% of clay on a weight basis. The mixtures were humidified using 10% water on a weight basis and formed using a uniaxial hydraulic manual press, with a mold of approximately 80x25x6 mm, and a pressure of 25 MPa for 30 s. In the beginning, the specimens were left to dry in the open air for 24 h, followed by oven drying at 110 °C for 24 h, and then heat treated in an electric oven at 1000 °C, with 1 h isotherm and 10 °C/min heating rate. The residue added in the clay mixture, and the temperature and sintering time used in this work were based on the best conditions obtained in previous work, with samples containing clay and 0, 20%, 40%, 60%, and 80% of ornamental rock residue, fired at 900, 1000 and 1100 °C for 1 and 2 h [6]. Other authors [10, 12, 15] also indicated this firing temperature/time (1000 °C/1 h) for red ceramics due to the resulting good physical and mechanical properties. Good mechanical properties were also obtained after durability testing at 900 °C, compared to ceramics sintered at lower temperatures [16]. In addition, 1000 °C is a consistent value compared to those used in industries that produce ceramic building materials.

After firing, the following technological properties were determined: water absorption (WA), apparent porosity (AP), bulk density (Bo), modulus of rupture (MR), and flexural strength (FS) while adapting the ABNT NBR ISO 10545-3 [17] and 10545-4 [18] standards to the size of laboratory specimens. To determine WA, AP, and Bo, the specimens were oven dried at 110 °C, followed by a thorough water impregnation and immersion. The parameters were calculated using the equations found in the ABNT NBR ISO 10545-3 standard [17]. The FS and MR were determined in a flexometer (Flexi 1000 LX-650, Gabbrielli). The modulus of rupture is the force necessary to break the specimen multiplied by the ratio distance between supports/width of the specimen and flexural strength is the value obtained from 3/2 of the modulus of rupture divided by the square of the minimum thickness along the fractured side of the specimen. In addition to these tests, linear shrinkage (LS) was determined by subtracting the length of the specimen before and after the firing divided by its initial length. The

results presented are the mean and standard deviation of the individual values obtained for a total of 10 specimens. Additionally, after the FS test, microstructural analysis of a cross-section of the specimens was performed in a scanning electron microscope (SEM) at different magnifications.

RESULTS AND DISCUSSION

The chemical compositions of the residues and clay used are shown in Table II. The results showed a difference between the chemical composition of silicate rock residues and marble residue and variability among the silicate residue samples as well. The SiO₂ content varied from 48.27% to 70.19% and the sum of Na₂O+K₂O+CaO+MgO ranged from 10.37% to 19.28% in the silicate residues; additionally, their loss on ignition (LOI) was relatively small, except for the Nero Marinace residue. The marble residue, on the other hand, had low SiO₂ (1.67%) and higher CaO (38.78%) contents, MgO content (15.83%) typical of carbonate rocks, and consequently higher LOI (42.46%) corresponding to the decomposition of the carbonate content.

The clay used to produce the ceramic mass was highly plastic, consisting of clay minerals from the montmorillonite and/or vermiculite and kaolinite groups, in addition to feldspar and quartz [12]. The mineralogical composition of residues (Fig. 1) from the silicate rocks indicated the presence of quartz (SiO₂), microcline (KAlSi₂O₂), plagioclase [(Na,Ca)Al(Si,Al)Si₂O₂], and $[(K,Na,Ca),(Al,Mg,Fe,Li)_{4.6}(Si,Al)_{8}O_{20}(OH,F)_{4}].$ mica Calcite was also identified in the Nero Marinace residue (corroborating the high LOI). In addition to quartz, mica, microcline, and pyroxene [(Ca,Na,Fe,Mg,Zn,Mn,Li) (Cr,Al,Fe,Mg,Mn,Ti,P)(Si,Al),O₄] were present in Verde Peacock while plagioclase, edenite [NaCa₂MgSi₇O₂₂(OH)₂] and enstatite $[(Mg,Fe)_{2}Si_{2}O_{4}]$ were observed in the Ocre Itabira sample. The Mármore Aquarela carbonate residue consisted mainly of calcite (CaCO₂) and dolomite $[CaMg(CO_2)_2]$. In the silicate residues, the K₂O came from K-feldspars and micas, while Na₂O was derived from both sodium feldspar and plagioclase. The CaO came mainly from the lime used in the sawing process on conventional looms, but plagioclase can also provide this mineral. In

Table II - Chemical	composition (wt%)) of the materials	used to prepare the cerai	mic mass (data cor	npiled from [5, 12]).
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Material	SiO ₂	TiO ₂	Al_2O_3	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P_2O_5	LOI
Crema Bordeaux ¹	70.19	0.05	11.87	6.66	0.06	0.05	2.97	3.34	4.01	0.01	0.81
Verde Peacock ¹	57.86	0.60	15.02	12.29	0.11	0.36	4.83	2.93	5.43	0.24	0.33
Ocre Itabira ¹	53.07	0.82	15.42	10.85	0.14	2.62	7.93	3.47	5.26	0.34	0.09
Nero Marinace ¹	56.45	0.32	8.28	14.32	0.18	0.67	10.25	2.03	1.57	0.11	5.82
Marrom Bahia ¹	51.16	1.21	14.77	15.59	0.25	1.19	5.95	4.95	3.91	0.70	0.32
Preto Aracruz ¹	48.27	3.06	14.00	13.89	0.16	4.41	8.64	2.58	2.26	1.81	0.93
Mármore Aquarela ²	1.67	0.02	0.65	0.51	0.01	15.83	38.78	0.01	0.03	0.04	42.46
Clay	59.14	0.95	24.12	7.79	0.08	2.90	1.03	n.d.	3.36	n.d.	n.d.

¹: silicate rock residue; ²: carbonate rock residue; LOI: loss on ignition; n.d.: not determined.

carbonate rocks, the CaO and MgO contents came from carbonates (calcite and dolomite) whereas iron oxide came from some minerals of the mica and plagioclase group that were present in some of the processed rocks, but mainly it came from the shot and steel blades used in the sawing process. Generally, the presence of alkali metals (such as K and Na) and alkaline earth metals induce the formation of a liquid phase during ceramic firing [10, 19]. Iron gives color and acts in the sintering process by reducing the viscosity of the liquid phase formed during firing, which can lead to pyroplastic deformation [12, 19]. In the burning process, attention is needed to the temperature for starting the liquid phase formation, which depends on the minerals formed by these chemical elements [8-13, 15, 16, 19].

This work investigated the effects of adding residues to

ceramic pieces fired at 1000 °C. The results of the physical tests performed with samples containing 80% clay and 20% residue on a weight basis are compiled in Table III. The physical properties of the specimens and the composition of the residues used to produce the ceramic mass were correlated, as shown in Figs. 2 and 3. The LS and BQ were noticeably lower in the bodies whose residues had higher LOI (Fig. 2a). In these materials, the LOI was associated with the carbonates present that during burning released CO_2 , leaving pores in its structure and possibly causing the part to expand. These pores decreased the density, as well as, occupied internal spaces in the samples, that were perceived by a smaller shrinkage during firing. Linear shrinkage is a property that indicates the densification degree during firing and is important for the dimensional control of the final



Mi=mica / Qz=quartz / Mc=microdine / PI=plagioclase / Px=piroxene Ca=calcite / En=enstatite / DI=dolomite

Figure 1: XRD patterns showing mineralogical compositions of the dimension stone residues used to prepare the ceramic mass.

Table III - Means and standard deviations of water absorption (WA), apparent porosity (AP), bulk density (Bq), linear shrinkage (LS), flexural strength (FS), and modulus of rupture (MR) of clay samples with added dimension stone residue.

Sample	WA (%)	AP (%)	BQ (g.cm ⁻³)	LS (%)	FS (MPa)	MR (N)
Crema Bordeaux	12.0±0.3	24.1±0.4	2.03±0.01	1.28±0.09	14.7±1.2	306±5
Verde Peacock	12.3±0.3	24.8±0.5	2.02±0.01	1.60±0.15	17.5±1.5	351±4
Nero Marinace	14.4±0.9	28.0±1.1	1.95±0.03	0.48±0.24	14.9±2.6	321±2
Ocre Itabira	9.7±0.5	21.3±0.8	2.18±0.02	1.34±0.20	17.6±2.2	353±4
Marrom Bahia	12.7±0.6	25.5±0.9	2.01±0.02	1.24±0.17	16.5±2.1	350±3
Preto Aracruz	11.4±0.4	23.7±0.6	2.08±0.01	1.56±0.24	16.9±1.7	343±3
Mármore Aquarela	16.7±0.3	30.6±0.4	1.83±0.01	-0.04±0.11	16.3±1.1	337±5

2.5

C (%) / **B** (6/cm³) 1.5 (%) / **B** (0.5 0.0 0.0

-0.5

a)

20

18

16

14

12

10

8

6

4

2

0

b)

WA (%) / FS (MPa)

2.03

0.81

Crema

Bordeaux

14.66

12.05

0.81

Crema

Bordeaux

2.02

1.60

(0.33)

Verde

Peacock

Linear Shrinkage (%)

17.54

12.26

0.33

Verde

Peacock

Water Absorption (%)

2.18

1 2

0.0

Ocre

Itabira

17.56

9.7

0.09

Ocre

Itabira

42.46

1.83

2.08

0.93

Preto

Aracruz

16.88

11.44

0.93

Preto

Aracruz

2.01

0.32

Marrom

Bahia

16.53

12.72

0.32

Marrom

Bahia

Flexural Strength (MPa)

Bulk Desnsity (g/cm3)

1.95

04

Nero

Marinace

14.37.93

5.82

Nero

Marinace

45

40

35

30

25

15

10

-5

-0

45

40

35

30

25

15

10

5

Mármore

Aquarela

--- LOI

20

%

-0.04

LOI

Mármore

Aquarela

42.46

16 đ

20 0

(%)

Figure 2: Relationship between linear shrinkage, LS, bulk density, $B\varrho$ (a), water absorption, WA, and flexural strength, FS (b), of ceramic pieces with the loss on ignition (LOI) of the residue.



Figure 3: Relationship between water absorption (WA), flexural strength (FS), and linear shrinkage (LS) of the ceramic pieces with SiO_2 and MgO+CaO+K₂O+Na₂O contents in the silicate residues with low percentages of carbonates.

product. Previous works [20, 21] relate that linear shrinkage values between 1.5% and 3.0% are recommended for red ceramics while values less than 1.5% are considered ideal. In this work, the silicate residue samples presented 0.48% to 1.83% shrinkage while carbonate samples expanded to -0.04%, both results within the recommended value. For the AIII ceramic group, the ABNT ISO 13006/2020 standard

[14] requires that the dimensions of any ceramic body stay inside the $\pm 1.5\%$ range of the mean obtained for 10 specimens. Nonetheless, the manufacturers of rustic porous ceramic tiles inform in their catalogs that these product sizes can vary. Therefore, according to such information, the measured linear shrinkage variations and their standard deviations did not disqualify the studied material for being used as this type of red ceramic.

Using residues with very high (Mármore Aquarela) and a relatively high (Nero Marinace) LOI resulted in ceramic bodies with higher WA values. On the other hand, flexural strength did not present the same relationship with the LOI, indicating that other factors interfered with this mechanical property (Fig. 2b). Research investigating the effect of adding 10% of carbonate rock residue (with more than 95%) of CaO) in clays used for producing rustic ceramic tiles [12] showed that the added residue increased both, WA and FS, while LS decreased. The increase in FS was probably due to the newly formed calcic solid phases [19]. Also, the SiO₂ content and the sum of alkali and alkaline earth elements and iron oxide affected the properties of the specimens that did not have high or very high LOI (Fig. 3). The sample containing the Crema Bordeaux residue had more than 70% of SiO₂, partially occurring as free silica, which damaged the sintering of the specimen; therefore, the WA value was intermediate, while the LS and mechanical strength were low compared to the other samples. Furthermore, in the silicate residue sample with lower SiO₂ levels (53% or less), the sum of fluxing oxides (Na,O+K,O+CaO+MgO) became decisive for densification. So much so that the sample containing the Ocre Itabira residue exhibited the lowest WA and the highest mechanical strength among all the analyzed samples (Fig. 3). According to ABNT ISO 13006 standard [14], ceramic tiles classified as AIII must have minimum mechanical strength values of 8 MPa, breaking load greater than 600 N, and WA greater than 10%. It is noteworthy that ABNT does not have specific values for rustic porous coating, but class AIII is the most appropriate category to evaluate this product type.

The MR values of all samples were lower than the minimum established in the ABNT ISO 13006 standard [14], and the FS values were above the ABNT requirements. In addition, the WA of the Ocre Itabira sample was lower than 10%, which classified this material in another class of ceramic tile regarding this parameter. The Crema Bordeaux residue had relatively lower percentages of alkali and alkaline earth



Figure 4: Images showing the aspects of the ceramic specimens made with clay and different residues sintered at 1000 °C.





Figure 5: SEM micrographs of ceramic pieces containing 80% clay and 20 wt% of the following residues: a,b) Crema Bordeaux; c,d) Ocre Itabira; e,f) Marrom Bahia; and g,h) Mármore Aquarela.

elements and higher SiO_2 content, thus indicating higher quartz and lower feldspars levels. Feldspars are known to form a liquid phase at eutectic temperatures. Thus, the lower levels of these minerals in the residue probably justify the lower FS and MR compared to the other samples. On the other hand, the Ocre Itabira residue had the highest levels of alkali and alkaline earth oxides, which generated greater densification and reduced the WA to a value lower than that of porous ceramic tiles type AIII.

Fig. 4 shows the difference in color and surface porosity of the samples containing different types of dimension stone residue. All samples were reddish while the sample with Ocre Itabira residue showed dark dots on the surface due to the higher Fe_2O_3 content from the steel shot used for rock sawing.

Fig. 5 shows the micrographs of sample specimens obtained by SEM at different magnifications, whose physical properties met the WA and FS requirements (clay and added Marrom Bahia and Mármore Aquarela residues), the Ocre Itabira sample with WA below the limit for porous ceramic tile and the Crema Bordeaux sample with lower FS and MR. All micrographs showed the typical porous structure of traditional clay ceramic, with a small portion of particles larger than 10 μ m and angular morphology. In the microstructure of the Crema Bordeaux sample, for example, some weakly bound regions indicated that sintering did not occur via the liquid phase, and others, with apparently well-cohesive particles, showed that fluxes had better performance. These microstructure characteristics

were evenly distributed throughout the sample. Despite the similar micrographs of the Ocre Itabira and Marrom Bahia samples, the micrographs at low magnification showed that the quantity of intergranular pores was lower in the first, consistent with the higher Bo (2.18 g.cm⁻³) and lower WA (9.75%) compared to the other sample $(2.01 \text{ g.cm}^{-3}, 12.72\%)$. The microstructure of the sample containing the Mármore Aquarela residue, in addition to residual intergranular pores from the sintering process, showed some porous channels probably formed by the CO₂ released during the calcination of carbonates present in the residue. The burning temperature of 1000 °C was enough to liberate the CO₂ present in the carbonates, leaving pores in the structure but not enough for the alkaline earth oxides (CaO, MgO) to actively participate in the liquid phase formation reactions, which would help the densification of the ceramic piece.

CONCLUSIONS

It was shown that the compositional variability of dimension stone residues from rocks sawed in the Cachoeiro de Itapemirim region (ES-Brazil) influenced the technological properties of rustic porous ceramic tiles. Besides the expected compositional differences between silicate and carbonate residues, from different rock types, there were relevant variations in chemical and mineralogical compositions within the group composed of silicate residues. Samples with higher carbonate contents and, consequently, higher loss on ignition (LOI), presented higher water absorption values, lower linear shrinkage, and lower bulk density, indicating lower densification. The microstructure of the specimens prepared with Aquarela Marble residue and clay confirmed the lower densification at the sintering temperature of 1000 °C for 1 h. In the silicate residue samples with low LOI, the SiO₂ content and the sum of oxides of alkali and alkaline earth elements and iron oxide affected the ceramic properties. The Crema Bordeaux residue had the highest SiO₂ content (>70%) among the studied samples. Part of SiO₂ occurred as free silica, which generated relatively lower linear shrinkage and flexural strength. The Crema Bordeaux sample showed heterogeneous microstructure regarding the densification. On the other hand, the highest fluxing oxides' content in the Ocre Itabira residue induced lower water absorption and higher flexural strength, indicating that the liquid phase generated during the sintering process was effective in wetting the particles and promoting good rearrangement and lower porosity in the ceramic. The dimension stone residues with intermediate SiO₂ content and fluxing oxide levels not very high were the most suitable for manufacturing a porous ceramic tile. Thus, separating different types of residues may give them greater added value when used as raw material.

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