Use of marble sludge waste in the manufacture of eco-friendly materials: applying the principles of the Circular Economy.

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Abstract

In order to apply the principles of Circular Economy, the suitability of a marble sludge generated in the ornamental rock industry of Andalusia (Spain) has been assessed as raw material in ceramic bricks. Mixtures containing a clayey-base and 0, 2.5, 5, 7.5 and 10 wt% of marble powder have been shaped into 60 × 30 × 10 mm^3 prismatic specimens and sintered at 950 °C in a muffle. The main technological properties of the bricks related to color, shrinkage, porosity, water absorption, suction and compressive strength have been determined. The addition of marble sludge has fostered the development of a lighter color, together with a significant increase of open porosity. This aspect has implied higher water-absorption and suction results and, on the other hand, a decrease of density and mechanical strength. The correlations obtained after applying Shapiro-Wilk normality tests and *r*-Pearson coefficients endorse the clear relationship between the addition of marble powder and the abovementioned effects on the technological properties of the sintered bricks. All the measured properties fully meet the brick's standardized requirements, which would indicate that the recycling of marble wastes could be a promising alternative to obtain eco-friendly lightweight ceramic materials.

1. Introduction

The currently preponderant lineal economic model is based on the sequence "take-extract, produce, consume and throw away". Nevertheless, the reckless use of material and energy resources has made it unsustainable from an environmental perspective [1, 2]. As a potential solution, the alternative "restorative" model of the circular economy aims not only to preserve resources, components and materials, but also to maximize their level of use by recovering, reusing and recycling strategies. Thus, the circular economy concept embraces a cycle of positive continuous development to protect natural capital, optimize returns on resources, minimize system risks and manage finite stocks and renewable flows. It is a model that works at any scale efficiently [3].
Ceramics sector offers an interesting playground in this aspect, because it allows the use of wastes as suitable raw materials, reducing the production costs too [4]. Among the residues to be used for this purpose, there is a vast body of literature about the use of ornamental rock sludge. Hence, most of the studies related to granite-marble sludge recycling as a component in ceramics are concerned with the manufacture of housing materials. Some examples are: red ceramics [5], porcelain tiles [6], bricks and ceramic tiles [4], bricks and floor tiles [7], ceramic bodies in general [8], roof tiles [9] and building bricks [10, 11]. These studies concur that the addition of proper proportions of rock sawing powder into the ceramic mixtures allows the production of ceramic bodies whose features may be even better than the commercial ones. Furthermore, the significant presence of alkaline and alkaline earth oxides in these materials was demonstrated to act as flux, reducing the firing temperature, and thus the process becomes more energy-efficient.
In the case of marble, its sector is booming in Spain, a country that is currently the seventh largest marble producer in the world and whose production is especially preponderant at the Eastern Andalusia area. The marble cutting industry has a worldwide production of around 236000 thousand tons whereby large enriched calcium-carbonate sludge volumes are generated. This sludge may lead to a negative environmental impact if it is not correctly managed [12].
The present investigation is focused on the reuse of a marble sludge to obtain ceramic bricks, analyzing its influence on the main technological properties as a function of the proportion of waste with respect to a clayey mineral base.

2. Materials and methods

2.1. Sampling and initial preparation of raw materials

Three clays from the ceramic industry of Bailén city (Jaén, Spain) have been used as major components: red clay (RC), blond clay (YC) and black clay (BC). Furthermore, a marble cutting sludge (M) has been employed as additive to study its influence on the properties of sintered ceramic bricks, especially on those related to pore formation. M was provided in an almost dry state by Centro Tecnológico Avanzado de la Piedra from the marble processing plants located at Macael region (Almería, Spain). The low water content of M (only 0.15 %) would mean a more efficient transport and handling, which is a very important aspect to save energy and costs.
A base-mixture of clay (here designated simply as C-0M; Table 1) was prepared according to the following percentages: 30 wt% RC + 30 wt% YC + 40 wt% BC. Once at laboratory, the samples were oven-dried for 24 h at 90 °C. The clays were ground in a Gruber® Duplex 21A hammer mill until obtaining a < 400 µm fine powder. The marble sludge did not require any milling-treatment because it presented a very fine particle size, as a result of the rock-sawing process whereby this residue is generated. Likewise, 0, 2.5, 5, 7.5 and 10 wt% of the powdered marble sludge (Fig. 1) was added to C-0M in order to conform the final mixtures intended to brick manufacturing. The name and composition of the mixtures are detailed in Table 1.

Table 1. Percentages and weighed quantities for the preparation of the different samples (grs.)

Mixture name	% M	Mass required for a 40 g specimen			
		M	YC	RC	BC
C-0M	0	0.0	12.0	12.0	16.0
C-2.5M	2.5	1.0	11.7	11.7	15.6
C-5M	5	2.0	11.4	11.4	15.2
C-7.5M	7.5	3.0	11.1	11.1	14.8
C-10M	10	4.0	10.8	10.8	14.4

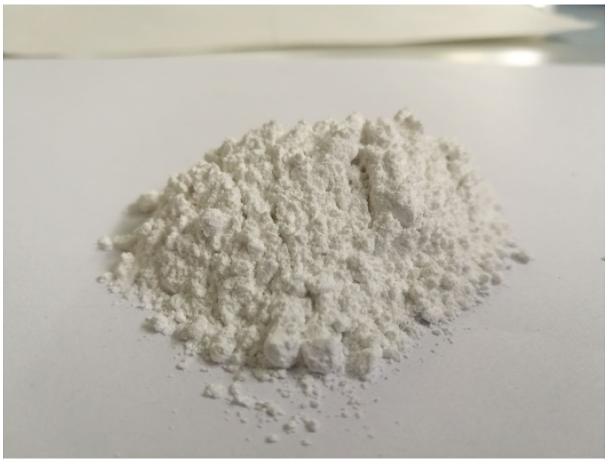

Figure 1 Marble sludge powder

2.2. Characterization of raw materials

The raw materials were characterized according to the following procedures:
The content of C, H, N and S was determined by elemental analysis using a CHNS-O Thermo Finnigan Elementary Analyzer Flash EA 1112 under O_2 atmosphere. The general chemical composition of M has been determined by X-ray fluorescence (XRF) through a Bruker's PIONER S4 EXPLORER equipment. The marble's organic matter percentage was determined by calcination at 430°C relying on the method of Navarro et al. [13]. The carbonate percentage of the marble sludge was obtained according to the UNE 103-200-93 standard [14].

2.3. Manufacturing of the bricks

First, the mixtures were blended thoroughly on dry state and then with 5.5 wt% of distilled water until getting a homogeneous mass, with a proper workability to obtain flawless pieces along the subsequent steps. Wet batches corresponding to 40 g of dry material were taken and shaped into prismatic specimens of 60 × 30 × 10 mm^3 by applying a load of 50 MPa with a uniaxial laboratory-type Mega KCK-30 A press. Then, the specimens were oven-dried for 48 h at 110 °C. Afterward, they were sintered in a Nabertherm LH 60/14 laboratory-scale electrically heated muffle at a heating rate of 5 °C/min up to 950 °C, maintaining this temperature for 60 min

[15]. After this time, the samples were left to cool by natural convection inside the muffle. The sintered bricks have been called according to the name of the mixture plus the term "B" (brick). For example: C-2.5M-B represents the brick specimens manufactured from the mixture containing 2.5 wt% of M. The external appearance of the sintered bricks is shown in Fig. 2.

Figure 2 Sintered bricks. Marble sludge increases from left (0 wt%) to right (10 wt%)

2.4. Characterization of the bricks

With regard to the sintered bricks, a complete characterization has been also performed:
The linear shrinkage of the green pieces and the sintered ones was calculated from the length of the samples before and after oven-drying and firing, respectively, according to EN 772–16:2011 [16], whereby measurements were taken with a precision of ± 0.01 mm using a caliper. The weight loss occurred after the green specimens being oven-dried was also determined. Similarly, the loss on ignition (LOI) during the sintering stage at 950 °C was worked out relying on the weight difference recorded before and after firing. Water absorption capacity was determined based on the guidelines indicated in the EN 772-21:2011 [17] and ASTM C373-18 [18] standards. Likewise, suction capacity was obtained through the standard procedure detailed in EN 772-11:2011 [19]. The bulk density of the specimens was determined according to the standard EN 772-13:2000 [20]. Compressive strength tests were conducted for the fired specimens based upon the EN 772-1:2011+A1:2015 standard [21]. A Suzpecar CME 200 SDC press was used for this purpose by applying a load of < 20 MPa/s on the brick's upper face until failure (Fig. 3). Apart from the abovementioned characteristics, the external appearance related to color has also been examined.

Figure 3 Application of the load during the compressive test on one specimen

2.5. Statistical analysis

The degree of interconnection between the variables related to manufacture-conditions and the technological properties of the sintered bricks was assessed through a correlation matrix. The software IBM SPSS Statistics v.24 was used for this purpose. First, Shapiro-Wilk normality tests were conducted in order to figure out the normality of the variables by considering a *p*-value of 0.05 as significance-level yardstick. Based on the normal or non-normal distribution of the data, Pearson or Spearman coefficients were employed to analyze the correlations, respectively. Correlations have been considered as significant when $p < 0.05$ and highly significant when $p < 0.01$ under bilateral analysis.

3. Results and discussion

3.1. Raw materials characteristics

Regarding the clays, it can be observed in Table 2 that BC holds greater carbon contents than the others (3.3 %). This measured carbon would be related to organic components, which are responsible for providing the black color, as well as of generating potentially pore-generator gases when burnt out. As displayed in Table 3, the percentage of carbonates of M was 100 % and the organic matter content was very low (0.1 %). Carbonates could be very positive to develop pore-generator-gases when this material is decomposed during firing. This agrees with its high carbon (namely inorganic) and CaO contents, which are 11.6 % and 55.4 %, respectively (Table 2 and Table 3). The LOI of 42.5 % is also an indicator of the meaningful susceptibility of this material to be thermally decomposed.

Table 2 Elemental analysis of clay and marble sludge

Raw material	% C	% H	% N	% S
YC	1.891	0.321	0.040	0
RC	1.036	0.459	0.045	0
BC	3.347	0.333	0.078	0
M	11.614	0.14	0.14	0.99

Table 3 Chemical and organic composition of the marble sludge

Raw material	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	LOI	Carbonates	Organic matter
M	0.32	0.14	0.14	0.99	55.36	0.24	42.47	100	0.1

3.2. Characterization of the bricks

According to Fig. 2, the color of the bricks (reddish) is influenced by the marble proportion, so that it turns lighter as the waste percentage is increased. About the technological properties, the results obtained from the bricks' characterization tests are summarized in Tables 4 and 5. Bearing in mind that contractions may lead to tension and cracks [22], bricks' linear shrinkage is an important parameter. While the green specimens shrank around 4-5 % when oven-dried (due to the weight loss associated to water evaporation, which was around 0.2 %), the firing stage involved a negative shrinkage in all the brick varieties, ranging between -0.15 % (C-0M-B) and -0.04 (C-10M-B). This implied a slight volume expansion in all specimens, which was less important when the marble sludge was added. From this perspective, it is assumed that all the specimens, and especially those containing M, are of good quality because the shrinkage is far from the 8 % yardstick, a value that ceramic pieces should not exceed according to Martínez et al. [23].

On the other hand, it is observed that the mass loss after firing or LOI (Table 4) tends to increase progressively with the marble-powder proportion, such that the percentage is 9.14 % when no waste was added, reaching 14.05 % with 10 wt% of this material. Hence, while the LOI of the base-mixture would be highly likely associated to the loss of structural water, the decomposition of organic matter and carbonates and the dehydroxylation of clay minerals, the application of marble has fostered an additional LOI due to the decomposition of the marble's calcium carbonate.

Table 4 Shrinkage and mass loss of the green and sintered bricks. Negative shrinkage means expansion. Data are expressed in percentage

Brick name	% M added	Oven drying shrinkage prior firing	Oven drying weight loss	Shrinkage after firing	Weight loss after firing (LOI)
C-0M-B	0	4.58	0.17	-0.15	9.14
C-2.5M-B	2.5	5.62	0.2	-0.05	10.03
C-5M-B	5	5.09	0.25	-0.06	11.14
C-7.5M-B	7.5	4.98	0.25	-0.07	12.24
C-10M-B	10	3.42	0.14	-0.04	14.05

Despite the LOI gain linked to the presence of marble waste, the shrinkage has barely changed, and indeed, it seems to decrease as explained above. This fact would point out that: *i*) the marble particles decomposition have fostered a small expansion of the material and *ii)* the spaces left by the marble particles when decomposed have not been closed during the subsequent sintering, so that they have become stable pores. This last aspect is in agreement with the open porosity data collected for each brick variety (Table 5 and Fig. 4a), in such a way that the porosity increases clearly as the added marble sludge proportion is higher (27.3 % of open porosity in C-0M-B against 36.8 % in C-10M-B).

Obviously, the enhancement in the open porosity has impacted directly on the rest of properties. In order to keep the same criteria followed until now, the results of C-0M-B will be compared to those of C-10M-B for representing the maximum and minimum ones, respectively. Thus, the addition of marble has implied a significant reduction in the bulk density from 1900 kg/m^3 to 1700 kg/m^3 (Table 5 and Fig. 4b), which not only could be very positive to lower the building load to the ground, but also to reduce the costs during transport.

On the other hand, the porosity's gain when M is added has also entailed greater absorptive capacities, as reflected the suction and absorption data in Table 5 and Fig. 4c,d, which increases from 2.3 % and 14 % in C-0M-B to 3.4 % and 20.2 % in C-10M-B, respectively. Although water absorption can affect negatively to ceramic pieces' durability [24], the bodies manufactured in this study meet the requirement stated in the EN 772-11:2011 standard [19] about not exceeding the threshold value of 4.5 kg/m^2·min in the suction test.

Table 5 Physical and mechanical properties of the sintered bricks. Data are expressed in percentage. Fc is compressive strength and d is bulk density

Brick name	% M added	Open porosity, %	Bulk density, kg/m^3	Suction, kg/m^2·min	Absorption, %	Compressive strength, MPa	Fc / d (× 1000), N/m·g
C-0M-B	0	27.3 ± 3.8	1900 ± 106	2.3 ± 0.7	14.0 ± 3.0	56.8 ± 1.2	29.9
C-2.5M-B	2.5	33.9 ± 0.1	1763 ± 3	3.1 ± 0.0	17.9 ± 0.2	53.8 ± 2.8	30.5
C-5M-B	5	34.1 ± 0.1	1742 ± 13	3.2 ± 0.1	18.3 ± 0.2	53.1 ± 2.0	30.5
C-7.5M-B	7.5	35.9 ± 0.3	1733 ± 8	3.3 ± 0.0	19.3 ± 1.4	50.8 ± 1.1	29.3
C-10M-B	10	36.8 ± 0.2	1700 ± 10	3.4 ± 0.0	20.2 ± 0.2	48.1 ± 0.7	28.3

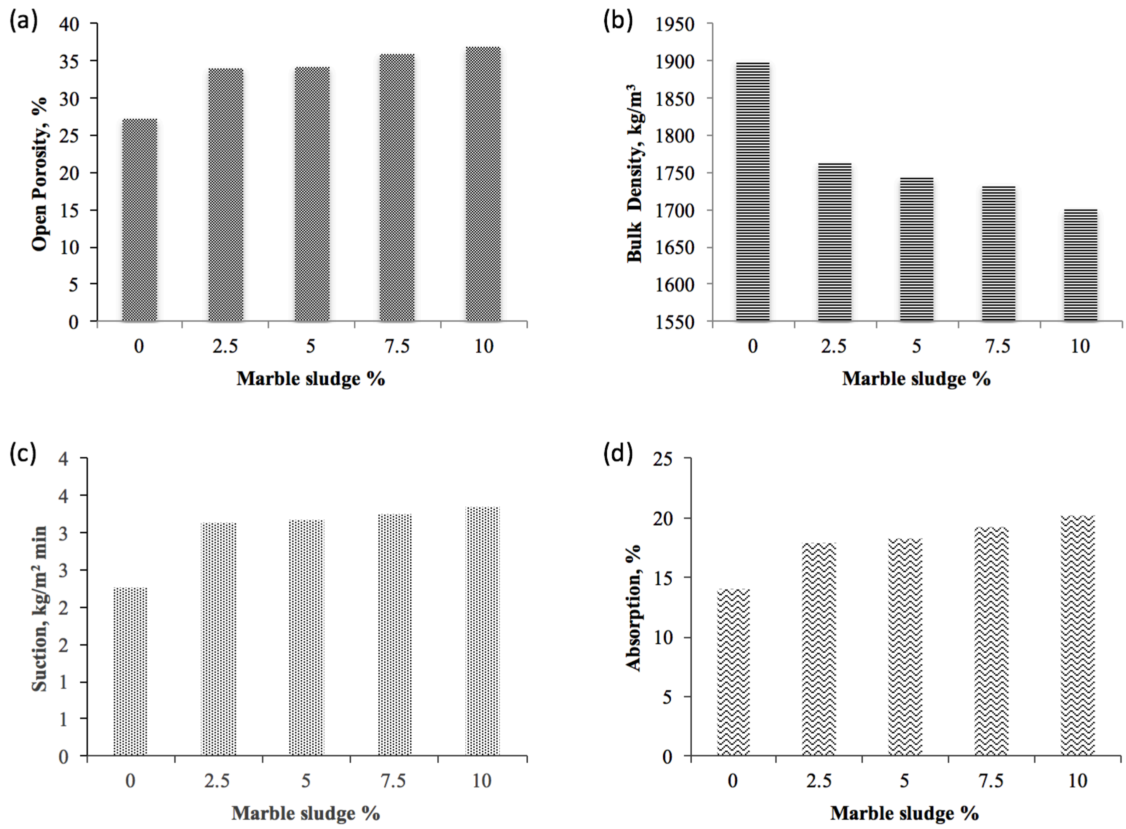

Figure 4 Graphical representation of the technological properties of the sintered bricks related to: (a) open porosity, (b) bulk density, (c) water suction and (d) water absorption

As expected, the pore formation tied to M-addition is directly connected with a gradual decrease of the compressive strength (Table 5 and Fig. 5), namely from 56.8 MPa to 48.1 MPa for 0 and 10 % of marble, respectively. Despite this, all the varieties fully compliant with the minimum value (10 MPa) required by the EN 772-1:2011 standard [21], so the use of marble sludge would be justified based on this fact. In addition, the ratio that relates the compressive strength and the density (last column in Table 5) would endorse this statement, because it hardly changes around 30 N/m·g regardless the proportion of M applied, which would denote that all the varieties exhibit a similar proportional mechanical strength.

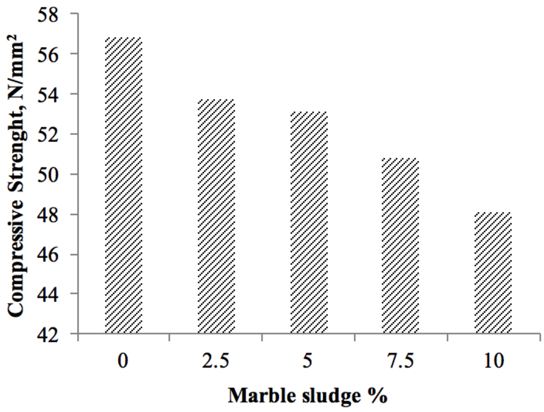

Figure 5 Graphical representation of the compressive strength evolution depending on the amount of marble sludge added

3.3. Statistics and relationship between properties

The statistical results about the normal distribution of the different variables measured are detailed in Table 6. As can be observed, it can be accepted the hypothesis of normality for all the parameters, since their *p*-values are greater than the reference ($p = 0.05$). This agrees with the high Shapito-Wilk statistic data, which are around 0.8-1.0.

Table 6 Shapiro-Wilk test results about normality. Normality is considered for *p*-values >0.05

Parameter	Shapiro-Wilk test	
	Statistic	*p*-value
% M added	0.987	0.967
Oven drying shrinkage prior firing	0.923	0.550
Oven drying weight loss	0.901	0.415
Shrinkage after firing	0.787	0.063
Weight loss after firing (LOI)	0.977	0.921
Open porosity, %	0.831	0.143
Bulk density, kg/m^4	0.820	0.116
Suction, kg/m^2·min	0.787	0.063
Absorption, %	0.879	0.306
Compressive strength, MPa	0.989	0.977
Fc / d	0.892	0.368

Therefore, considering that the data extracted from the studied parameters follow a normal distribution, Pearson's *r*-coefficient can be applied to study the correlations between them (Table 7). Although a general discussion of the relationship between variables was elaborated in previous sections, the analysis of the *r*-values enables a quick and accurate way to demonstrate the degree of interconnection between them. Thus, the

correlations displayed in Table 7 show that there is a strong dependence between the addition of marble (% M) with the increase of LOI during firing, the porosity and the associated water absorption (r = 0.9-1.0), but on the other hand, affecting the compressive strength very negatively (r = -0.986). As was noted in previous sections, this would be related to the formation of pores, so that, if we focus on the correlations of the open porosity with the rest of parameters, it can be detected easily that this property is linked significantly to the increase of shrinkage after firing (r = 0.933), the suction capability (r = 0.994), the water absorption (r = 0.997), and the drop of compressive resistance (r = -0.910).
An interesting relationship is detected when facing the shrinkage when oven-drying the green material (R_{drying}) and the compressive strength / bulk density ratio (Fc/d), such that this one seems to increase as the former does. It could involve that under similar conditions of sample-preparation (percentage of water added and load applied by the press to conform the specimens), those materials that exhibits larger shrinkage values prior to firing could lead to an enhanced mechanical resistance with respect to density than those whose shrinkage is much reduced.

Table 7 Pearson's r correlation matrix relating the measured parameters under bilateral analysis. In bold those r values that are significant for $p < 0.05$. In bold plus asterisk (*) when r is significant for $p < 0.01$. Symbols: % M= % M added; R_{drying} = Shrinkage after oven-drying; LOD = loss of weight on oven-drying; R_{firing} = Shrinkage after firing; LOI = Loss on ignition (weight loss after firing); P = Open porosity; d = Bulk density; Suc = Suction; Abs = Absorption; Fc = Compressive strength

Parameter	**% M**	**R_{drying}**	**LOD**	**R_{firing}**	**LOI**	**P**	**d**	**Suc**	**Abs**	**Fc**	**Fc/d**
% M											
R_{drying}	-0.567										
LOD	-0.032	0.707									
R_{firing}	0.720	-0.040	0.133								
LOI	**0.991***	-0.668	-0.164	0.678							
P	**0.891**	-0.181	0.189	**0.933**	0.842						
d	-0.878	0.183	-0.194	**-0.953**	-0.832	**-0.991***					
Suc	0.864	-0.128	0.250	**0.948**	0.809	**0.994***	**-0.997***				
Abs	**0.918**	-0.247	0.148	**0.923**	0.876	**0.997***	**-0.992***	**0.990**			
Fc	**-0.986***	0.562	0.125	-0.772	**-0.985***	**-0.910**	**0.893**	-0.876	**-0.933**		
Fc/d	-0.750	**0.884**	0.543	-0.209	-0.815	-0.435	0.386	-0.356	-0.481	0.760	

4. Conclusions

Relying on the circular economy principles, the inclusion of a large scale residue (marble sludge) as a technological raw material in the ceramic industry has been carried out. Different proportions ranging 0 – 10 wt% of marble powder waste have been added to a mineral-base comprised of a ceramic clay mixture to be sintered into bricks.
The technological properties of the obtained specimens point out that these could be perfectly used in construction for complying with the different standards related to bricks.
The marble sludge addition has entailed a significant increase of the open porosity as a consequence of the calcium carbonate decomposition that the marble undergoes during firing. The porosity's gain has been accompanied by a reduction of the bulk density and an increase of water absorption and suction capability, but meeting the standard requirements in any case. About the former, the lower density could involve cost and energy savings related to the transport and the placement of these materials, as well as a reduction on the building load to the ground.
Likewise, the addition of marble sludge has entailed a certain loss in compressive resistance due to the presence of a more porous structure. However, all the specimens have yielded a very good mechanical behavior (around 50 MPa), such that the compressive strength of the least resistant brick variety (48.1 MPa) is significantly higher than the minimal value required (10 MPa).
All the experimental results have been analyzed statistically, in such a way that the correlations obtained after applying Shapiro-Wilk normality tests and r-Pearson coefficients endorse the clear relationship between the addition of marble powder and the abovementioned effects on the technological properties of the sintered bricks.
In conclusion, it has been demonstrated that the recycling of marble wastes can become an excellent alternative in the production of lightweight eco-friendly bricks.

Acknowledgments

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