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Physico-chemical, mineralogical, and technological characterization of stabilized clay bricks for restoration of Kasbah Ait Benhadou- Ouarzazate (south-east of Morocco)

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ABSTRACT

Due to ageing, the unfired brick masonry may detach and fall off, or deteriorate to such an extent that it becomes necessary to restore them. Such is the case, for instance, of the Ksar of Ait Benhadou in Ouarzazate, Morocco. Our study aims to provide compatible and sustainable earthen bricks to restore this monument. Samples were collected from facades that were under conservation/restoration at the time when sampling was performed.

Clayey soil samples were collected vicinity to the Ksar and analyzed by X-ray diffraction and X-ray fluorescence. A representative sample was stabilized with three aggregates (lime, cement and straw). The effect of ageing of the stabilized briquettes on shrinkage, water absorption, mechanical and thermal properties, compressive strength and thermal conductivity was studied on stabilized specimens. The samples consist mainly of clay minerals, calcite and quartz. They are rich in iron, aluminium and potassium. These samples are sandy with low plasticity (PI = 7%), which is slightly lower than the plasticity value required by the Moroccan standard for earth constructions. The results showed that the stabilized clayey soils have suitable properties such as density, porosity, water absorption and high thermal insulation. The best compressive strength performance is obtained for clay-stabilized samples with a high sand fraction. The thermal conductivity of clay-stabilized specimens increases as the specimens become denser and more compact, lime and straw have the opposite effect.
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1. Introduction

The alarming consequences of global warming pose a challenge to the global growth model, which has so far been driven by profit and gain alone, without considering the environmental impacts. Among the most energy-consuming sectors in Morocco is the building construction industry, this sector consumes up to 33% of the country's total energy expenditure, with 7% of tertiary buildings and 26% for residential buildings [1].

Morocco has set a target of reducing CO_2 emissions by 13% by 2030 in order to address this global problem, and thus control and reduce greenhouse gas emissions, raw earth building materials can help reduce energy consumption and preserve the environment [2].

Some non-exhaustive advantages of earthen construction include the availability of earth, it's almost limitless recyclability, the economic effectiveness of construction techniques, hygroscopic and thermal qualities, and the occupant's health [3–5].

Earthen architectural heritage may be found on all inhabited continents, with earthen materials used in the construction of 20% of the architectural structures on the UNESCO World Heritage

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Fig. 1. Panoramic view of the Kasbah of Ait Benhadou[1].

List. On ecological, social, economic and cultural levels [6,7], this style of construction is unquestionably beneficial.

The majority of research on clays used as construction materials concentrated on stabilizing them with lime or cement [8–12].

For the short-term reaction, referred to the initial lime consumption, about 5% of lime was sufficient, also, the lime addition improves soil qualities by reducing plasticity and swelling [8]. Stabilization with a mixture of 3.5% cement (LAFARGE, Saint Pierre Lacour) and 3.5% lime (industrial quicklime, PIGEON Saint Pierre Lacour 35) achieved the highest strengths, after 28 days the unconfined compressive strength (UCS) is being 3 MPa compared with 2 for 7% lime and 2.3 MPa with 7% cement. The Californian Bearing Ratio (CBR)at 7 days was 31%, six times higher than of the unstabilized material [9]. In addition, unfired clay bricks behave favorably in most building applications and present an environmentally friendly alternative to fired bricks [10].

Since 1987, the Ksar Ait Ben Haddou (Fig. 1) has been listed on the UNESCO World Heritage List. This building attracts tourists from all over the world. Further, it is also known as a cinematographic site, and it knew the shooting of several masterpieces such as Gladiator, Kingdom of Heaven etc. To which it owes its worldwide fame. Since the early 1990, the Ksar has been a subject of a series of restoration interventions by Centre for the Restoration and Rehabilitation of the Architectural Heritage of the Atlas and Sub-Atlas areas (CERKAS to preserve the heritage and environmental components of the site. The intervention was funded by the Moroccan Ministry of Culture, United Nations Development Program (UNDP) and UNESCO technical assistance [13].

The goal of this research is to characterize the archeological earthen material of Ksar ait benhaddou's wall in order to reuse the wall debris as a viable restoration material. Hydraulic binders (lime and cement) and vegetable fibers of straw will be added to stabilize earthen material sampled from the Ksar Ait Benhadou wall debris. The small amount of the adjuvants (5%) will increase the mechanical and thermal performance of earthen materials. The study also includes the investigation of the effect of aging or pre-curing for 3 days to favor the mixing. The pre-curing is an ancestral technique used in the field of earthen buildings as well as in the design of ceramics by the local inhabitants of the region. Zhang et al. [14] provides a detailed overview of the different pre-cure and cure prototypes adopted by several researchers and their impact on bricks from waste materials, which is an original approach.

2. Materials

Three clayey samples, E1, E2, and E3, were collected from the debris of the Ksar Ait benhadou's outer wall located near north-west of the city of Ouarzazate, southeast of Morocco (Fig. 2). The extraction points' coordinates were obtained using Google Earth program (Table 1).[1]

A representative sample of earthen material was mixed with water and stabilized with various proportions of cement (DC), lime (DL) and wheat straw (DS) (Table 2). Cement (CPJ 30) and slaked lime were purchased on the local market, and wheat straw was obtained from local producers. The percentage of straw added was limited to 3% because of its lightness. In fact, if it is used in a large quantity, the clay fraction will be drowned in the straw [15]. The sample is mixed with water to form a paste and then pre-cured in plastic bags in order to favor the mixing for 3 days. We have adopted the pre-curing of the mixtures before shaping brick specimens firstly because the small amount of hydraulic binder (<5%). This content did not modify the plasticity of the clay and caused fractures in the microstructure of the hardened mortar. Secondly to avoid the evaporation of the mixing water due to the high temperatures in the region allowing clay particles to consolidate. Then, brick specimens of the size of $8 \times 4 \times 1.8$ cm^3 were prepared from aged pastes to determine the optimum content of mixing water and the influence of additives on setting. For uniaxial tests at room temperature, brick specimens are cubic in shape with the size of $5 \times 5 \times 5$ cm^3 [16].

3. Experimental methods

The particle size analysis was carried out on the debris of earthen material by sieving the fraction greater than 80 μm [17] and by a sedimentometry method for the fraction <80 μm [18].

The mineralogical composition of the clayey bulk samples was determined by X-ray diffraction (XRD) technique using a Shimadzu 6100 diffractometer, equipped with an X-ray Cu tube, operating at 40 kV and 30 mA, in the range of 2θ [2–70°], with a step size of 0.02° and scanning time of 1 s per step.

Oriented aggregates prepared from the fraction less than <2 μm were carried out on a glass slide. The XRD is recorded between 2 and 30°, using the same conditions as for bulk measurements. These oriented aggregates were subjected to three successive treatments, air drying, glycolation, and heating to 500 °C for 4 h [19,20].

The chemical composition was obtained by X-ray fluorescence, using the Axion spectrometer, with wavelength dispersion of 1 kW.

The Atterberg limits of the fraction less than 400 μm were determined using the Casagrande apparatus on the cured pastes [21].

The specific surface was determined using the methylene blue method [22], the total volume of the methylene blue added (VBS) allows to calculate the specific surface of sample debris [23,24].

The calcium carbonate content of the debris sample was estimated using the Bernard calcimeter method [25].

The organic matter content of the debris sample was obtained from the loss on ignition method at 550 °C for 4 h [26].

The Proctor test allows determining the optimum mixing water content [27]. The optimum water content of stabilized specimens was determined from the curve of the dry density as a function of the volume of water [28,29].

The mechanical resistance tests were carried out on stabilized specimens using a TESWELL machine with a capacity of 20 KN and a speed of 0.5 mm/min.

4. Results and discussion

Earthen materials from the wall of the Kasbah Ait Benhadou are composed of 56% of particles smaller than 200 μm. They consist of

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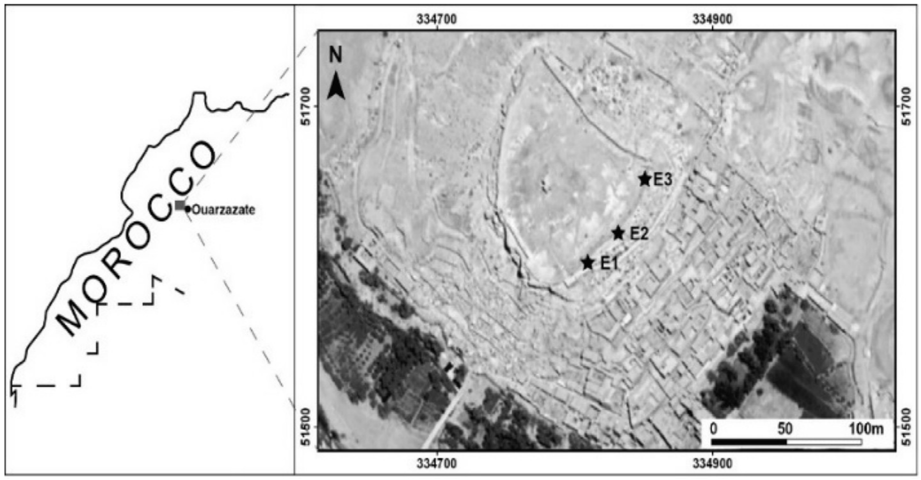

Fig. 2. Location map of the sampling area.

Table 1
Coordinates of samples studied.

Samples	Latitude	Longitude
E1	31° 16′ 55, 17″ N	4° 16′ 12, 82″ W
E2	31° 16′ 53, 20″ N	4° 16′ 12, 31″ W
E3	31° 16′ 51, 96″ N	4° 16′ 11, 96″ W

Table 2
Nature and amounts of aggregates used to manufacture brick specimens.

Brick specimens	Debris (%)	Cement (%)	Lime (%)	Straw (%)
EC1	99	1	0	0
EC2	98	2	0	0
EC3	97	3	0	0
EC4	96	4	0	0
EC5	95	5	0	0
EL1	99	0	1	0
EL2	98	0	2	0
EL3	97	0	3	0
EL4	96	0	4	0
EL5	95	0	5	0
ES1	99	0	0	1
ES1.5	98.5	0	0	1.5
ES2	98	0	0	2
ES2.5	97.5	0	0	2.5
ES3	97	0	0	3

79.77% of sand (>80 µm), 16.86% of silt (2–80 µm) and 2.94% of clay (<2µm) (Fig. 3). Based on to these results, the clay sample was classified according to the McManus diagram, (Fig. 4). These materials exhibit moderately high porosity and medium permeability according to this classification [30]. According to the literature, the particle size distribution of raw materials directly affects the properties of the final manufactured bricks [24,31].

Chemical composition shows the predominance of calcium (48.5 wt%), silicon (18.8 wt%), and iron (16.3 wt%). Titanium and other metals are present in very low amounts (Table 3).

The mineralogical composition shows the presence of quartz, calcite, dolomite, clay minerals (chlorite, illite and palygorskite) (Fig. 5).

Since the peak at 7 Å disappears when heated to 550 °C, indicating the occurrence of kaolinite is present in the clay phase. The XRD results of the clay fraction confirm the presence of chlorite (d≃7.12 Å) and illite (d≃10 Å) and palygorskite (d≃10.50 Å) (Fig. 6).

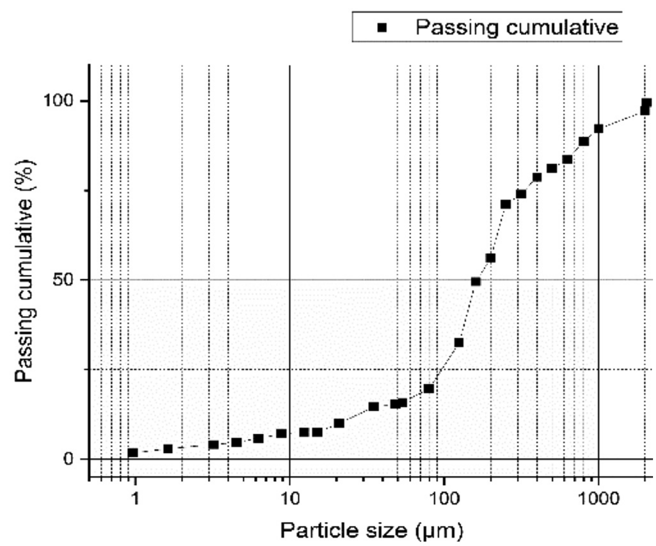

Fig. 3. Particle size distribution of raw clay sample.

The geotechnical results of the examined sample (Table 4) show moderate plasticity according to the Burmister and Atterberg classification [21,32].

The liquid limit (LL) and the plasticity index (PI) are slightly below the lower limit of the Moroccan standard regarding the seismic regulation for earthen constructions, on the other hand, the value of the plasticity limit is included in the acceptable range of the same Moroccan standard (25 < LL < 45% and 10 < PL, PI < 25%) [16].

The proportion of organic matter content is 3.8%, which is a little higher than the recommended value [27,33].

Carbonates content is 10.3%, which is mostly calcite, classifying it as a moderately calcareous soil according to the norm NF P 94–048 [25].

The determination of the specific surface area (SSA) by the methylene blue, does not allow to have the exact value because it does not take into account the microporisity and also the interaction with the adsorbent material. The presence of swelling minerals shows a significant increase in specific surface area when measured by the MB [23]. The soil we used, rich in kaolinite, does not contain swelling clay. For kaolinite and kaolinitic soils, the SSA values obtained by methylene blue and N2 adsorption are close to

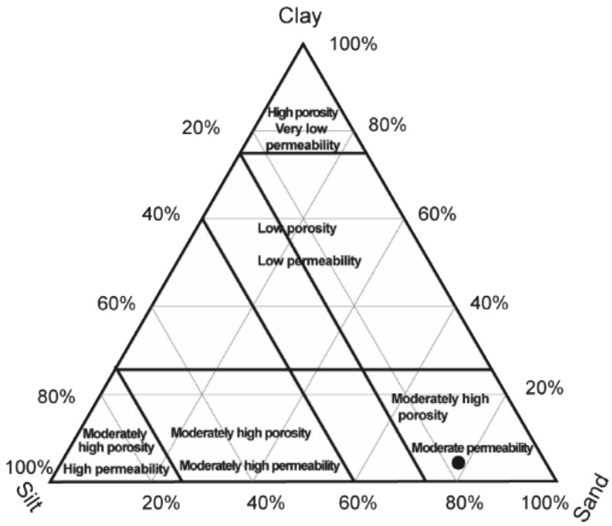

Fig. 4. Position of the debris from the wall of the Kasbah.

30 cm^2/g [23]. For the soil we studied, the SSA determined by the methylene blue method is 30.5 cm^2/g. This value is close to those determined for soils of similar composition, which can vary from 11.3 to 27.8 m^2/g [34]. In general, the methylene blue method widely used by researchers when they do not have the possibility to use the BET, to have an approximate value of the SSA [35–37].

\The scanning electron microscope results of the studied debris confirm the dominance of the finest fraction (1 to 3 μm) and a rough texture. The presence of such a texture ensures strong particle cohesiveness after the addition of a binder, which is beneficial for improving the mechanical performance of the stabilized earthen materials (Fig. 7). Small particles are cohesive that tend to agglomerate [38].

The accent of cementation depends on three mechanisms [39]: (1) the mechanical keying of the hydration products of cement with the rough surface of aggregate (often covered with fine cracks); (2) the epitaxial growth of hydration products at some aggregate surfaces; (3) the physical-bchemical bond between the hydrating cement paste and the aggregate, due to chemical reaction.

The optimum liquid/solid ratio, determined from mortars density, was found equal to 20.3% (Fig. 8).

5. Characterization of stabilized earthen debris

Compressive strength is one of the most important tests to be able to evaluate the quality, and the suitability of the clayey materials for load bearing walls [40,41].

The compressive strength values of the samples after 28 days, are in accordance with the minimum values accepted by the Moroccan standard of 1.17 MPa (Fig. 9) [16].

The optimum quantity of cement for most soils is between 6 and 12%; this hydraulic binder is indeed very favorable for the stabilization of sandy soils with low plasticity indices <20%, as in our case [42,43].

As mentioned above for cement, the optimal percentage of lime recommended to stabilize the soil varies from 6 to 12% [44,45]. Lateritic soils stabilized with lime have shown compressive strength values between 1 and 1.4 MPa [46,47].

As expected, the compressive strength value of the specimens, stabilized with cement, increases linearly with the percentage of the added cement. Cement gives the bricks a better consistency and therefore a better compressive strength. In addition, the high sand content in the earth debris contributes to the consolidation of the hardened specimens.

We found that the obtained compressive strength values are lower in comparison to those reported in some previous studies [46–49], the results show that compressive strength of stabilized specimens with cement and lime are in accordance with the minimum values acceptable by the Moroccan standard of 1.17 MPa, but those stabilized with wheat straw are lower than the lowest limit, and therefore rejected [16].

Table 3
Chemical composition of debris from the wall of the Kasbah Ait Benhadou.

	Ca	Si	Fe	K	Al	S	Mg	Ti	Sr	P	Cu	Pb	V	Cr	Zn	Rb
%W	48,5	18.8	13.4	8.3	3.3	2.9	1.9	1.4	0.2	0.1	0.1	0.07	0.06	0.06	0,005	0,04

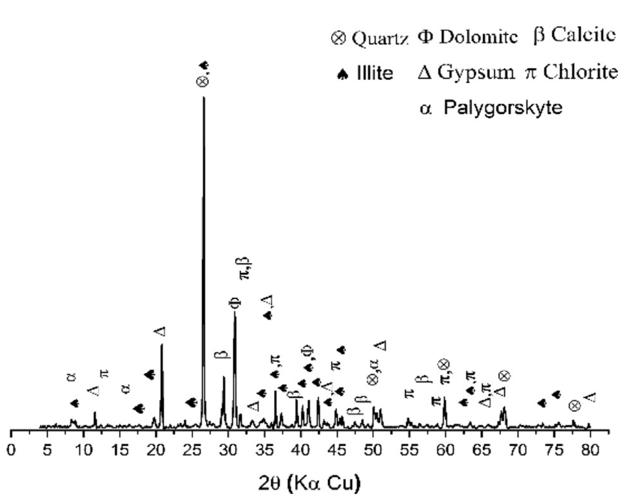

Fig. 5. X-ray diffraction diagram of debris sample from the wall of the Ksar Ait Benhadou.

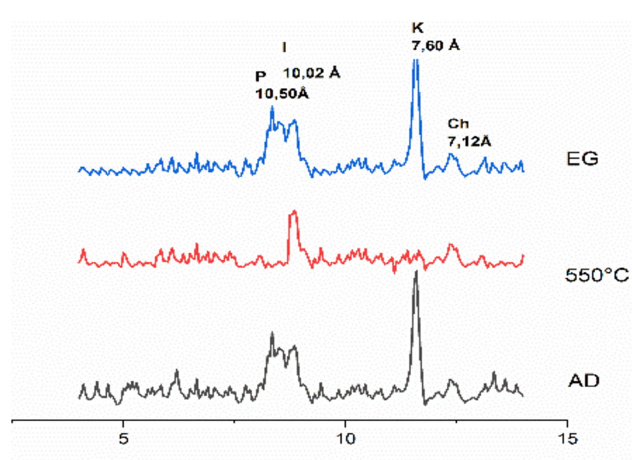

Fig. 6. X-ray diffraction diagrams of the clay fraction (<2 μm) of debris from the wall of the ksar Ait Benhadou. A: air dried; EG: glycolation; heating at 550 °C. (Ch: Chlorite, I: Illite, K : Kaolinite).

Table 4
Physical and geotechnical characteristics of earthen material from the Kasbah Ait Benhaddou.

Properties		sample material
Atterberg limits (%)	Plasticity limit	16%
	Liquid limit	23%
	Plasticity index	7%
Total organic matter (wt.%)		3.8%
$CaCO_3$ (wt.%)		10.3%.
Volume of methylene blue (g/100 g)		1.46
Specific surface area (m^2/g)		30.5

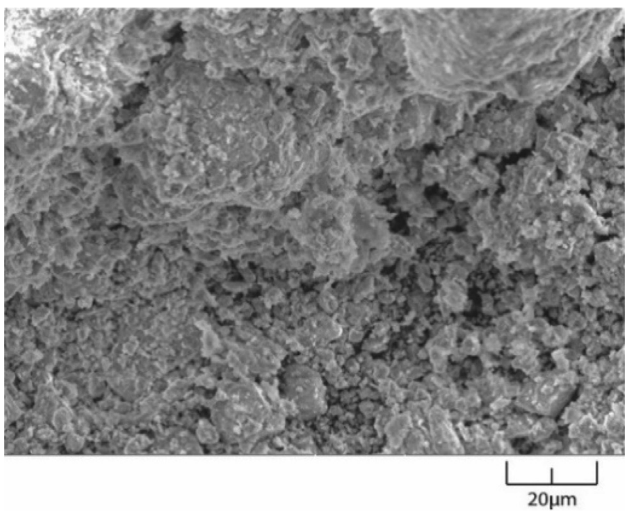

Fig. 7. SEM image of earthen material debris from the Ksar Ait Benhadou.

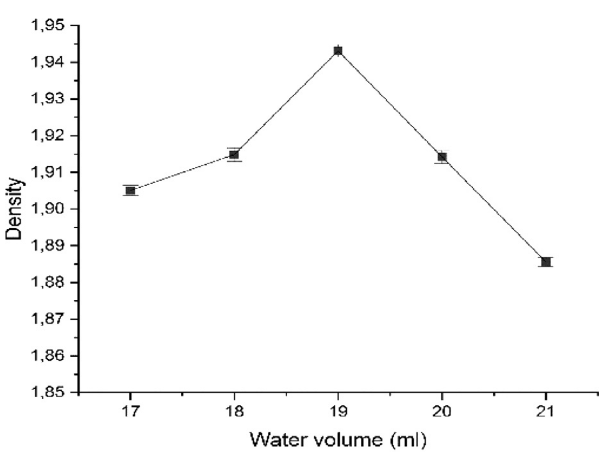

Fig. 8. Evolution of the density of the stabilized material according to the content of the mixing water.

The low value of compressive strength revealed for our stabilized specimens by cement and lime compared to other studies [50], is presumably due to the techniques used for curing. In the outdoors, the dry cure does not ensure enough humidity necessary for pozzolanic reactions, liable for the consolidation of the fabric, as compared with the wet cure.

With the aim of enhancing the strength characteristics of clays, Sunil Kumar Meena et al., [51] have used wheat straw. The optimum fiber content they found in their study is about 1.0%, which is similar to the optimum fiber content of other types of natural and synthetic fibers. In our work, we found that the optimum percentage of straw is about 2%.

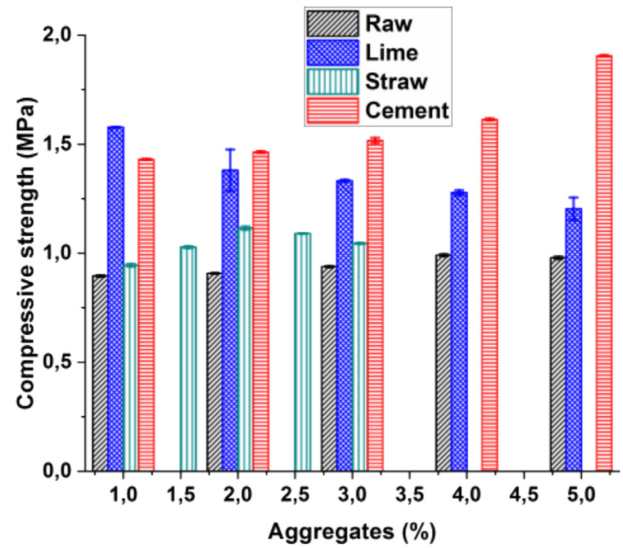

Fig. 9. Compressive strength of reference sample and stabilized specimens.

6. Conclusion

In this study, we have characterized samples of debris from the wall of the Kasbah of Ait Benhaddou and then we have stabilized them with cement, lime, and straw in order to improve the mechanical and possibly thermal performances.

The Ksar Ait Benhadou's debris consists mainly of quartz, calcite, dolomite, chlorite, and illite. The debris sample exhibits a medium plasticity, a minor percentage of non-swelling clay, and a high proportion of sand. The choice of these additives did not come arbitrarily. In fact, the hydraulic binders, especially the cement, improve the mechanical resistance of the specimens, considering that our sample is essentially sandy. Moreover, the recycling of the straw has a double goal:

- to decrease the thermal conductivity by creating voids and lowering the points of contact, which causes the reduction of the weight of the bricks.
- to minimize the discharge of straw into the cultivated fields, especially as the climate of the region is dry and arid in the summer and characterized by long periods of heatwave (temperatures can reach 47 °C).

Cement with a high sand fraction produces good compressive strength.

CRediT authorship contribution statement

M. Lechheb: Conceptualization, Data curation, Writing – original draft. **A. Harrou:** Methodology, Data curation. **G. El boukili:** Data curation, Methodology. **M. Azrour:** Supervision, Conceptualization, Formal analysis. **A. Lahmar:** Investigation, Visualization, Formal analysis. **M. El Ouahabi:** Writing – review & editing. **E.K Gharibi:** Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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