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# The effect of non-toxic industrial wastes on the performance of fired earth bricks used in the rehabilitation of old buildings in Skikda, Algeria

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**ABSTRACT:** This study examines the recycling of industrial waste into fired bricks for use in building rehabilitation, inspired by research that reports benefits to masonry quality and the environment. To this end, bricks from the old town of Skikda (Algeria), were analysed from a mineralogical, physical and mechanical point of view and compared with new bricks realized with clay from Mila (Algeria) mixed with different industrial wastes. Namely the clay mixtures were prepared with 10% sand and optimal amounts of sawdust (WS), steel filings (SF), fine marble powder (FMP) and very fine marble powder (VFMP), with substitution rates of 5%, 20%, 20% and 20%, respectively. Except for bricks with WS, which showed reduced resistance (-46.95%), the FMP, SF and VFMP bricks showed satisfactory mechanical performance with compressive strengths of 9.7, 9.8 and 11.1 MPa respectively. Moreover, the leaching analysis indicates that they are environmentally friendly.

**KEY WORDS:** Quarry sand; Wood sawdust waste; Steel filings waste; Marble waste; Fired bricks.

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**RESUMEN:** *El efecto de los residuos industriales no tóxicos sobre el rendimiento de los ladrillos de tierra cocida utilizados en la rehabilitación de edificios antiguos en Skikda, (Argelia)* . Este estudio examina el reciclaje de desechos industriales en ladrillos cocidos para su uso en la rehabilitación de edificios, inspirado en investigaciones que reportan beneficios para la calidad de la mampostería y el medio ambiente. Para ello se analizaron desde el punto de vista mineralógico, físico y mecánico ladrillos del casco antiguo de Skikda (Argelia) y se compararon con nuevos ladrillos realizados con arcilla de Mila (Argelia) mezclada con diferentes desechos industriales. En concreto se prepararon mezclas de arcilla con un 10% de arena y cantidades óptimas de aserrín (WS), limaduras de acero (SF), polvo fino de mármol (FMP) y polvo muy fino de mármol (VFMP), con tasas de sustitución del 5%, 20%, 20%. y 20%, respectivamente. Excepto los ladrillos con WS, que presentaron una resistencia reducida (-46,95%), los ladrillos FMP, SF y VFMP mostraron un rendimiento mecánico satisfactorio con resistencias a la compresión de 9,7, 9,8 y 11,1 MPa respectivamente. Además, el análisis de lixiviación ha indicado que no liberan elementos nocivos para el medio ambiente.

**PALABRAS CLAVE:** Arena de cantera; Residuos de serrín de madera; Residuos de limaduras de acero; Residuos de mármol; Ladrillos cocidos.

## NOMENCLATURE

*Abbreviations:*
*SOFB:* Skikda old fired bricks;
*CMB:* Control fired bricks;
*FMPB:* Fired bricks with fine marble powder;
*VFMPB:* Fired bricks with very fine marble powder;
*SFB:* Fired bricks with steel filings;
*WSB:* Fired bricks with sawdust;
*MC:* Mila clay;
*FMP:* Fine marble powder;
*VFMP:* Very fine marble powder;
*SF:* Steel filings;
*WS:* Wood sawdust;
*Sand:* Sigues Sand;
*Indexes / Ratios:*
*PL:* Plastic limit (%);
*LL:* Liquidity limit (%);
*PI:* Plasticity index;
*SE:* Sand equivalent (%);
*Indissoluble %:* Resistant to attack and non-soluble particles.
*Symbol:*
*M:* Specimen weight (g);
*V:* Sample volume ($cm^3$);
*R:* Shrinkage (%);
*HCl:* Hydrochloric Acid;
*PH:* Hydrogen potential pH-value;
*D:* Maximum particle diameter (mm);
*B:* Volume of injected blue solution ($cm^3$);
*m0:* Mass of the organic matter (g, kg);
*AW:* Water absorption ($kg.m^2.min$) ;
*F:* Firing shrinkage (%);
*D:* Dry shrinkage (%);
*R:* Total linear shrinkage (%);
*WL:* Weight loss (%);
*DM:* The capillarity absorption (%);
*P:* Total open porosity (%);
*Test abbreviations:*
*(XRF):* X-ray fluorescent;
*(XRD):* X-ray diffraction;
*(MBV):* Methylene Blue Values (g/kg);
*(DTA-TG)*: Thermogravimetry Differential Thermal Analysis;
*(SEM):* Scanning Electron Microscopy;
*(TCLP):* Toxic Characterization Leachate;
*Greek Letters*
$\gamma_s$*:* Bulk density (g/cm) 3;
*Yd:* Max compaction conditions, Proctor normal test ($t/m^3$);
*C Strength:* Compressive strength (MPa);
*T Strength:* Tensile strength (MPa);

## 1. INTRODUCTION

Algeria, has valuable historic centres characterized by fired bricks architecture dating back several centuries. Notably, colonial architecture also made extensive use of fired bricks, but these are now in bad conditions of conservation (1, 2). In view of this situation, a few rehabilitation projects have started (3, 4), but they have not been successful, due to the difficulties encountered in selecting alternative materials, and to ignorance of the specific performance criteria requested for the new materials (5). This article describes the need to study carefully the fired bricks of the historical structures and the problems of the unavailability of compatible fired bricks for replacement. According to Azil et al. (6) and Mishra et al. (7), these structures require the production of suitable materials (8), an objective achieved through corrective interventions, combining the traditional process with innovative techniques, and the manufacture of low-cost alternatives instead of expensive industrial ready-to-use products, which are harmful to our heritage. As a case study for the use of these new bricks with additives for rehabilitation purposes, the buildings on Rue Didouche Mourad in the old town of Skikda, on the north-east coast of Algeria, were selected. The district in which Rue Didouche Mourad is located was founded in 1839 by the French government on the Roman ruins of the ancient town of Stora (9), according to the European architectural model used in the new towns of the Maghreb. Integrated studies to rehabilitate old buildings in the town of Skikda, including 27 buildings on rue Didouche Mourad (3), carried out by CTC (Technical construction control) Annaba and EPSEB-UPC (Escuela Politécnica Superior de Edificación de Barcelona- Universitat Politècnica de Catalunya) in Barcelona in 2016, confirmed that most of the buildings' walls are made of fired bricks, while the floors are made of metal beams and fully fired brick vaults as well as arches (Figure 1). Currently, the buildings are affected by numerous decay phenomena, such as walls that have lost mass or show deep cracks (Figure 2).


**Figure 1**. Didouche Mourad Street: a) in the past and b) currently.


**FIGURE 2**. State of conservation of the buildings: a) destroyed brick kiln; b) destroyed brick vault floor; c) deterioration of brick walls resulting in cracks; d) use of cement; e) presence of salt efflorescence, dark moisture and vegetation growth; f) infilling of a void with unsuitable bricks.

With the aim of producing compatible and better quality fired bricks, several studies were carried out on the integration of industrial waste as a substitute in different ceramics (10). As shown in (11, 12), clay correction has always been carried out to obtain suitable and inexpensive fired bricks with waste. As explained below, the research focuses on the possibility of restoring historic fired brick structures with innovative and compatible bricks obtained by adding industrial waste to the clay raw material. It is a multidisciplinary approach, combining theory and technology. It responds to current issues, particularly economic and environmental (8, 13). The fact that the industrial sector in Algeria produces an enormous quantity of waste each year (statistically, it is estimated at more than 2,500,000 t/year) motivated this choice (14-16). The recycling of this waste, a real threat to nature and public health, concerns the Algerian authorities (17). Recycling waste from the ceramic industry remains a useful and effective solution to the depletion of natural resources and the saturation of industrial landfills (18). Saving natural resources and energy, protecting the environment and public health, and reducing construction costs are the main advantages (19).

The behaviour of fired bricks depends on the type of clayey minerals used and the manufacturing process (20, 21). In fact, the composition and the grain size of the clay together with the transformation process determine the physical and mechanical characteristics of the fired bricks that are obtained. This explains why it is necessary to look for a good quality clay, composed mainly of silicates, with a moderate amount of calcite and iron oxide (22). Numerous studies, such as those by Baglioni et al. (23) and Călătan et al. (24), state that the clay must quite rich in clayey minerals fraction and have a minimum amount of organic material. Within the clayey minerals fraction, a high amount of kaolinite should be desirable rather than swelling clay minerals which presence can be dangerous because they cause excessive shrinkage during drying (25). Another problem can be the excessive presence of sulphates, which leads to the formation of soluble salts (26). However, the clay can be corrected by a degreasing process, as it has been experimented in many research works based on strategies for the integration of local materials, used as suitable ecological and economic alternatives for construction purposes, as proved by Shakir et al. (27). Aneke et al. (28), in their studies on the use of industrial waste for marginal construction materials, showed a considerable improvement in fired brick strength and a decrease in absorption capacity from the addition of dolomite, silica fumes, river sand and fly ash. Other researchers have investigated the recycling of various kinds of waste, like the incorporation of bagasse ash with alumina sulphate (29), green and core sand, fine sand (30-31), waste glass (32), ceramic powder, marble dust and wood ash (33), oat husk and barley husk (34), post-treated river sediments, lightly contaminated harbour and reservoir sediments mixed with fly ash (35), and demonstrated significant improvement in the physical and mechanical properties and sustainable performance of fired bricks. On the contrary, researchers such as Dai et al. (36) confirmed that the use of tannery sludge, sewage sludge, textile sludge and paper sludge worsen the physical properties of the fired bricks.

In the present work, the restoration method adopted is initially based on detailed laboratory characterisation of ancient materials, a source of useful information on construction techniques, raw materials and fired brick manufacturing technology. In order to identify our material, a multi-scale characterisation of the old bricks in the old town of Skikda was carried out. This involved determining their mineralogical, physical and mechanical properties. The main results will initially be used to characterise the behaviour of old bricks, but will also provide data for the production of new bricks (10, 14, 37, 38). As a next step, the following four materials, already tested in ceramic stabilization, are investigated as addition to Mila clay from East Algeria for the production of fired bricks to be used in the rehabilitation: calcareous sand, marble powder (MP), wood sawdust (WS), and steel filings (SF). Indeed, calcareous rock crushing stations in Algeria generate large quantities of residues such as limestone sand, which constitute both an environmental nuisance and a waste of raw material. The idea of exploiting it in the stabilization of fired bricks remains interesting in terms of economic and environmental benefit (39). The marble powder was selected because it is widely available as a result of the huge Algerian marble industry (40). Rasool et al. (41) and Prakash et al. (42) noted that it can react favourably with the earth's clay minerals. Indeed, calcareous ceramics are present from Neolithic to Roman age (11). The wood sawdust is a waste from the construction industry and pollutes the environment because it is often landfilled or burned in the open air (15, 43). Moreover, as reported by other scholars such as Uchechukwu et al. (44), its use as an additive could offer an environmentally friendly alternative for the production of bricks with specific physical properties. Its use reduces the need for its disposal and preserves non-renewable clayey minerals resources. The addition to the clay increases the porosity of the fired bricks without causing shrinkage. The steel filings, as heavy metals, are dangerous for human health, and their disposal is often difficult. To help solve this problem, researches have been conducted demonstrating that it is possible to recycle up to 20 % by weight of steel dust in fired brick production (45-47).

To achieve our objective, reference to high production standards is mandatory (48). Previous studies have reported that good quality fired bricks are hard, free from cracks, chips and large lime particles, such as that of Attar (49). Taha (50) reports that fired bricks of good quality are hard, free of cracks, splinters, and coarse particles of lime and that a suitable amount of clayey minerals matrix, a correct drying process and an angular shape of the framework grains reduces stresses that can cause cracks and shrinkage. Fired bricks in the ASTM standard (29, 51) require a value of shrinkage along with the firing process of less than 8%, and water absorption and porosity rates lower than 20% and 40%, respectively. The compressive strength should be between 6.9 and 27.6 MPa, according to NR EN 771-1/CN (52). Finally, to assess the possible environmental impact of the selected waste additives on the fired bricks, the leaching of heavy metals using the Toxicity Characterisation Leachate (TCLP) method (US-EPA 1311) (53, 54) was verified.

The performance evaluation has been carried out in laboratory by means of compositional, physical and mechanical analyses in order to study the reactions that develop between the additives and the clayey raw material. Thus, thanks to the role of research laboratories, it is possible to envisage effective solutions for the manufacturing of suitable fired bricks to be used in rehabilitation.

## 2. MATERIALS AND METHODS

### 2.1. Test characterization

#### *2.1.1. Characterisation Skikda old fired bricks*

Our methodology was inspired by previous research work, in accordance with a complementary test protocol (10, 14, 37, 38). The Bulk density and true densities were determined according to the ASTM C67-17 (55). The porosity accessible to water was determined by hydrostatic weighting according to the NF ISO 501 and ASTM C20 standards (56). The moisture properties were determined by the capillary absorption method according to NF EN 771-1+1A/CN (57). The mineralogical composition was determined by XRD (X-ray diffraction) (X'Pert PRO diffractometer by PANalytical equipped with X'Celerator detector and HighScore software for data acquisition and interpretation. The operating conditions were as follows: Cu $K\alpha 1$= 1.545Å radiation, 40 KV, 30 mA, $2\Theta$ = 3-70°) (56). In addition, the degree of hydraulicity was determined by thermal analysis (DTA-TG) using a thermogravimetric analyser (TGA PERKIN ELMER). The heating ramp was set at 5°C/min from initial temperature (20°C) to final temperature (1200°C). The mechanical properties were assessed by the compressive strength using the uniaxial compression test with the grinding and bonding method according to (58) and EN 771-1/CN (59). The tensile strength was measured according to NR standard (EN 771-1/CN) (59), (XP P 13-901, 2001) based on the split tensile test (Brazilian test) (6, 60). Finally, the surface morphology of the fired clay bricks was evaluated by SEM observation (JCM-5000 NeoScope) in accordance with ISO 22493: 2008 (En) (61), with metallization of the samples, with a fine layer of gold (62).

*2.1.2. Raw materials characterisation*

Preliminary analyses to characterise Mila clay (MC) and the additives “sand and powder of marble, wood sawdust and steel filings” were carried out in order to meet the compatibility criteria:

- The chemical composition of Mila clay and additives was determined using an X-ray fluorescent (S2PUMA-BRUKER) spectrometer (EDXRF) analyser (XRF).
- The mineralogical composition was determined through an X’Pert PRO diffractometer by PANalytical equipped with X’Celerator detector and HighScore software for acquisition and interpretation of data according to the following operative conditions: CuK α1= 1.545Å radiation, 40 KV, 30 mA, 2Θ = 3-70°) (56).
- Geotechnical characteristics: the density was measured with a pycnometer, according to XP CEN ISO/TS 17892-3 (63), the moisture content was measured in accordance with the standard XP CEN ISO/TS 17892-1 (64), the amount of organic matter was determined as per XP P94-047 (65), the shrinkage limit as required by DIN EN 1015-1 (66). Assessment of the optimum moisture content and compaction conditions was based on the Proctor test according to the standard NF P94-093 17 (67).
- The determination of the grain size distribution was carried out in two stages: sieving according to P 18-560 (68) and by sediment analysis according to XP CEN ISO/TS 17892-4 (69).
- The Atterberg limits were measured using a Casagrande apparatus, while the plastic limit test was determined on rolled thread, according to NF P94-051 (70) and the XP CEN ISO/TS 17892-12 (71) standard. The sand equivalent was determined according to the standard EN 933-8 (72).
- The specific surface area was determined by the methylene blue index (MBI) method according to standards NF P94-068 (73) and NF ENP 933-9 (74).
- Soluble salts were evaluated according to standard EN 1744-1 (75).
- In addition, the lime content was determined by attack with HCl, according to Belgian standard NBN 589-209 (76) and the degree of hydraulicity was determined by thermal analysis (DTA-TG).

*2.1.3 Characterization of repair bricks*

The following analyses were carried out in the new repair bricks:

- The Bulk density was calculated using Equation [1] according to ASTM C67-17 (55). After drying in an oven at 110°C until complete dehumidification, the weight was measured and divided by the apparent volume. The bulk density was calculated using the equation [1]:

$$Bulk\ Density\left(\frac{g}{cm3}\right) = \frac{M}{V} \quad [1]$$

- Critical shrinkage and weight loss were measured in accordance with (30, 62). Five samples were evaluated and their average value was reported. Using a calliper and a scale calibrated by measuring wet and firing weights, as well as initial length (L1), oven-dried length (L2) and final length (L3) after firing.

The drying shrinkage and weight loss were calculated from Equations [2], [3], [4], [5]:

$$Firing\ shrinkage\ (F) = \frac{L2 - L3}{L2} \times 100 \quad [2]$$

$$Dry\ shrinkage\ (D) = \frac{L1 - L2}{L1} \times 100 \quad [3]$$

$$Total\ linear\ shrinkage\ (R\%) = D + F \quad [4]$$

$$WL\%\ Weight\ loss = \left(\frac{(Original\ Weight\ -\ Fired\ Weight)}{Original\ Weight}\right) \times 100 \quad [5]$$

- The capillarity absorption was carried out according to NF EN 771-1+1A/CN (57). The process requires the determination of the area "A", Section submerged in water in $m^2$, then the difference between each weight M and the initial mass Mo was calculated per unit area:

$$DM = ((M - M0)/A) \times 100 \quad [6]$$

Then this measure is a function of the square root of weighing time, the water absorption coefficient AW expressed in (kg.m$^2$.min), the margin of tolerance <6, (AW) was calculated using, Equation [7]:

$$AW\ (kg\,.m2\,.min) = \left(\frac{\Delta Mt - \Delta M0}{\sqrt{t}}\right) \quad [7]$$

- The open porosity was determined by hydrostatic weighing according to the NF ISO 501 and ASTM C20 standards (56). The bricks were dried in an oven at 110°C until dehumidification. This requires an oven and a hydrostatic balance. After obtaining the initial mass (M0), the emerged mass (M1) and the quenched mass (M2) (g), the open porosity was calculated using Equation [8].

$$\%\,Porosity\ = \frac{(M2 - M0)}{(M2 - M1)} \times 100 \quad [8]$$

- The mechanical properties were evaluated through the compressive strength of the fired bricks by means of a single-axial compressive test using the grinding and bonding method according to EN 1926/1999 (58) and EN 771-1/CN (59). Using the Tinius Olsen Universal Hydraulic Test Machine, Super L, to test critical materials up to 3000 KN. The compressive strength (F, MPa) was calculated using Equation [9]:

$$C\ strength\ (MPa) = \left(\frac{F}{A}\right) \quad [9]$$

- The tensile strength was measured according to NR standard (EN 771-1/CN) (59), (XP P 13-901, 2001) based on the split tensile test (Brazilian test) (6, 60). Using the Tinius Olsen Universal Hydraulic Test Machine. Equation [10], below was used to compute the tensile strength (F, MPa) of the fired samples:

$$T\ strength\ (MPa) = 0.9 \times \left(\frac{2F}{\pi \times l \times h}\right) \quad [10]$$

Five samples were tested for each test, and the average of the results was taken as the final result.

- Determination of the amount of calcium carbonate through the Dietrich-Frühling calcimeter according to the NF P 94-048 standard (76). The method consists of determining the volume of carbon dioxide ($CO_2$) released by the action of an excess of HCl (at known temperature and atmospheric pressure) on a sample taken for testing.
- Moreover, in the new fired bricks the surface morphology was observed by electron microscopy (JCM-5000 NeoScope) according to ISO 22493: 2008 (En) (61), on gold metallized samples (62). This is done by scanning the surfaces with an electron beam and collecting the resulting image. The gold metallization is necessary to increase the conductivity and thus improve the qualification of the image.
- Toxity Characterization Leachate: this characterization was necessary to assess the environmental impact of the new repair bricks because the chemical characterisation of the raw materials showed a significant presence of heavy metals in some of them. The heavy metal leaching was determined through the Toxic Characterization Leachate (TCLP) method (US-EPA 1311). The leach test is limited to the following heavy metals: chromium (Cr), manganese (Mn), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As), cadmium (Cd) and lead (Pb), as shown in Table 13. The results were compared with US EPA (1996) limits for heavy metal concentrations (53, 54). Regarding the test protocol, the samples were crushed and sieved using a 0.7 µm glass fibre. The standard liquid was prepared for 1 mol of NaOH and HCI. Two extract fluids were then prepared. Extract 1 had a pH of 4.93, followed by the addition of glacial acetic acid and NaOH to the water; extract 2 had a pH of 2.88, followed by the addition of glacial acetic acid with water without NaOH. The sample was then prepared in polyethylene screw bottles, leaching at a ratio of 1.20, shaking at 30 rpm for 18 hrs. The leachate was filtered through 0.7 µm glass fibre filters and analysed using a PinAAcle 900 H/PerkinElmer atomic absorption spectrometer for the determination of dissolved metals (54).

## 2.2. Materials characterization

*2.2.1. Skikda old fired bricks*

Skikda old bricks were taken from the buildings during the rehabilitation works, in Didouche Mourad Street (Figure 3). These bricks show a red colour with dimensions of 22x11x5 cm. In Table 1, the physical and mechanical characteristics of the Skikda fired bricks are reported. The bulk density is 1.74 g/cm$^3$, porosity is 31%, water absorption is equal to 8.79 kg/m$^2$ min, compressive strength is 4.3 MPa and the tensile strength 1.7 MPa. These results reveal that the Skikda fired bricks are too fragile and do not meet the requirements of the NR EN 771-1/CN standards (62). This may be due to the effects of the decay suffered by the bricks over time, otherwise, the fired bricks were only used for “non-load-bearing” filling. The mineralogical analysis through X-ray diffraction shows the presence of quartz, microcline, mullite and hematite (Figure 4). These two last minerals are formed during the firing of a clay low in calcium carbonate. Their presence suggests a firing temperature between 800 and 900 ºC (77). Finally, the TGA curve of the Skikda fired bricks (Figure 5) shows a loss of weight of 11% which can be explained by the loss of moisture at 110°C, loss of "bound" water at 200-250 °C, and then the dihydroxylation of other minerals like muscovite (77, 78). The 87.93% of the sampled remained undecomposed.

**Figure 3**. Bricks taken from Didouche Mourad Street.

**Table 1**. Physical and mechanical characteristics of Skikda old fired bricks.

| Sample | $\gamma_s$ (g/cm$^3$) | AW (kg/m$^2$.min) | P (%) | T strength (MPa) | C strength (MPa) |
|---|---|---|---|---|---|
| SOFB | 1.74±0.02 | 8.79 ±0.36 | 31.0±0.1 | 1.7±0.3 | 4.3±0.4 |

Note: (SOFB= Skikda old fired bricks; $\gamma_s$ (g/cm) $^3$ = bulk density; AW (kg/m$^2$.min) = Water absorption;
P (%) = open porosity; T strength (MPa) = Tensile strength; C strength (MPa) = Compressive strength);

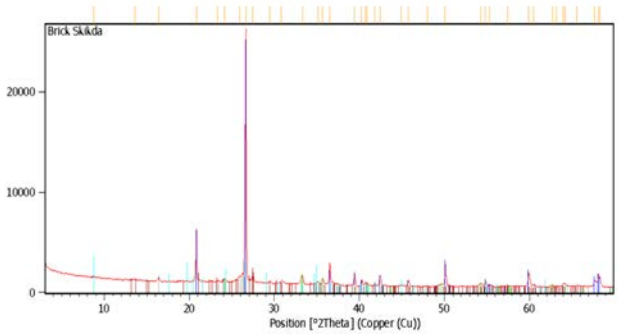


**Figure 4.** X-ray diffraction patterns of Skikda old fired bricks.

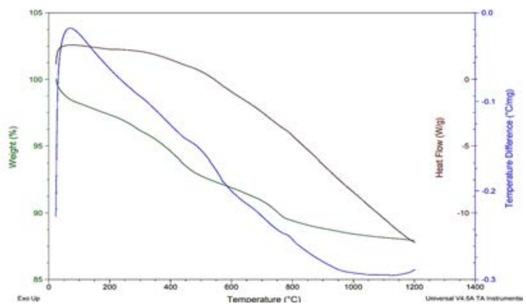


**Figure 5**. Thermogravimetric analysis of Skikda fired bricks.

*2.2.2. Mila clay*

The clay used to produce the new fired bricks was taken from a quarry situated near Mila, a city of north-eastern Algeria. Geologically, the site is characterized by a shale formation dating from Miocene to Quaternary (79). This clay was selected because it is widely used in eastern Algeria by craftsmen in bricks and ceramic production. The sampling was carried out at depths of 50 cm and more, extracted with a mechanical excavator (Figure 6). The clay in its natural state contains a certain amount of humidity (depending on the season and weather conditions) and presents three unmixed fractions of different colours, red, grey and brown (Figure 7). The analyses were carried out on a clay sample selected by quartering, a method used when large amounts of material are involved (80).

**Figure 6**. Extraction of the clay from Mila by an engine.

**Figure 7**. Texture of Mila clay in natural state.

*2.2.2.1. Chemical characteristics*

The major elements composition (Table 2) is characterized by a high amount of CaO and a considerable amount of $SiO_2$. Traces of Cl are found together with a certain amount of sodium and potassium that can help to improve the resistance of the material as it promotes melting phenomena, as described in the literature (22). The presence of heavy metal elements in Mila clay was found in different proportions according to the waste. The lowest percentage in the composition is copper Cu, followed by Ni, Rb and Sr, Zn, Cr, Zr, Mn, which are 0.022%, 0.03%, 0.03%, 0.037%, 0.05%, 0.07%, 0.08%, 0.15% respectively.

Heavy metals, such as arsenic (As) have not been detected. Particular care should be taken with heavy metals. The concentration detected should not exceed US EPA regulations (53).

**Table 2**. The results of the chemical analysis of Mila clay.

| Sample | CaO% | $Al_2O_3$% | $Fe_2O_3$% | $SiO_2$% | MgO% | $Na_2O$% | $K_2O$% | Cl-% | $SO_3$% | Mn % |
|---|---|---|---|---|---|---|---|---|---|---|
| MC | 24.76 | 16.00 | 16.48 | 50.20 | 1.96 | 1.36 | 2.94 | 0.04 | 0.33 | 0.15 |
| Sample | Ni % | Cu % | Zn % | Rb % | Sr % | Cr % | Zr % | V % | Pb% | |
| MC | 0.03 | 0.02 | 0.05 | 0.03 | 0.37 | 0.07 | 0.08 | 0.08 | 0.03 | |

Note: MC= Mila clay;

*2.2.2.2. Mineralogical characteristics*

In Figure 8, Tables 3 and 4, the principal mineralogical composition and the mineralogical composition of the clayey minerals fraction (below 2µm) are reported, respectively. The presence of calcite in an amount of 15–20% allows the clay to be classified as a slightly marly clay (79). This mineral, during firing at a temperature of 750–800 °C, is transformed into calcium oxide (CaO) (81) that, in presence of silica and alumina coming from the destruction of the clay minerals lattice, reacts to form calcium silicates and aluminates (78). Traces of dolomite and plagioclase are present. As for the clayey minerals, kaolinite is the most abundant, but the presence of a certain amount of swelling clay minerals (smectite and illite-smectite) can give shrinkage problems during drying. For this reason, this clay needs an adequate grain size correction and particular care during the bricks production cycle (27).

**Table 3**. Principal mineralogical composition of Mila clay (semi-quantitative data).

| Portion | Calcite | Quartz | Plagioclase | Dolomite | Clay minerals |
|---|---|---|---|---|---|
| MC | 18 | 20 | tr | tr | 62 |

**Table 4**. Mineralogical composition of the clay portion (semi-quantitative data).

| Portion | Kaolinite | Illite | Illite-smectite | Smectite | Chlorite |
|---|---|---|---|---|---|
| MC | 45 | 10 | 20 | 15 | 10 |

*2.2.2.3. Physical characteristics*

Figure 9, Figure 10 and Table 5 show the physical characteristics of the Mila clay. The index of plasticity is 23.4, indicating an average plasticity (12, 50). The grain size distribution shows the presence of 50% of clayey minerals, indicating a clay with a too high amount of clayey minerals. The shrinkage is considerable, at 7.7 %. MBV=5.13 confirms that the clay is cohesive, sticky, with a modular texture in the wet state. According to these data, researchers (48, 82) assert that a correction of the grain size distribution is essential for bricks production. On the other hand, the content of organic matter as well as the amount of soluble salts is not harmful, as confirmed by (66). The Proctor test confirms that the Mila clay is rich in clayey minerals, with a dry density =1.73 t/m3 and an optimal water content equal to 12.16 % (25). These data delimit the amount of water to be used when preparing the clay mixture, in order to obtain the maximum density for the minimum amount of water (83). Mila's $CaCO_3$ content of 20 % means that Mila's clay already contains a large amount of calcite, which is further increased by additives.

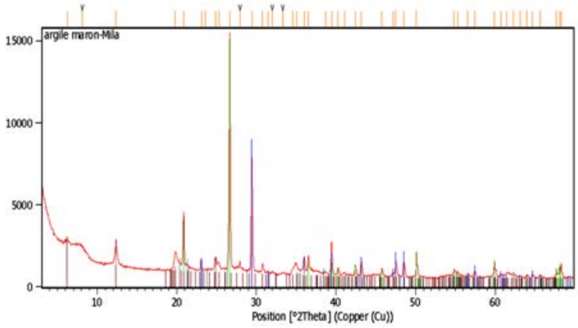


**Figure 8**. X-ray diffraction patterns of Mila clay.

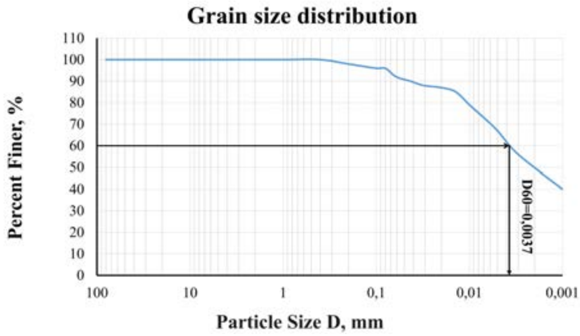


**Figure 9**. Grain size distribution.

**Table 5**. Physical characteristics, grain size distribution, soluble salts.

| Characteristic | Mila clay |
|---|---|
| **Liquidity limit (%):** | **LL= 48.98** |
| **Plastic limit (%):** | **PL= 25.58** |
| **Plasticity index:** | **PI= 23.4** |
| **Grain size (%):** | **14 (sand), 36 (silts), 50 (clay)** |
| **Methylene blue values:** | **5.13** |
| **Water content (%):** | **4** |
| **Density: kg/m $^3$ (%):** | **25.15** |
| $CaCO_3$ % | 20.00 |
| Indissoluble % | 63.20 |
| Organic matter content (%): | 5 |
| Salt content, LCTP/% Gypsum Group Procedure (Ca $SO_4$, $2H_2O$) (%): | 0.92 |
| Chloride (%): | 0.18 |
| Linear shrinkage (%): | R= 7.7 |
| Proctor Normal Test: $Y_{d\,Max}$ (t /m$^3$), % $W_{opt}$ (%). | $Y_{d\,Max}$=1.73, $W_{opt}$ = 12.16 |

The thermogravimetric curve of Mila Figure 11 shows a total loss in weight of the raw material estimated at 18.57%, which means that 83% of the clay was left undecomposed. Peak values are recorded at 110 °C, with a loss of 2.97% due to moisture, and between 200 and 250 °C, due to loss of "bound" water or to the loss of "hydrated" intercalated cations (as in the swelling of clayey minerals). Gypsum also exhibits endothermic effects in the range of 120 - 160 °C (77, 78). The 1.96% lost in weight between 550-650°C, is associated with losing organic matter. This is also the temperature range in which kaolinitic clayey minerals can be identified by its relatively strong endothermic activity (62, 82) and the 7.3% mass loss at 750°C - 850°C following the decomposition of well-crystallized CaCO3 (34, 62, 82).

*2.2.3. Waste additives*

- The methodology used for sampling remains a preparatory phase carried out before characterizing and valorising the waste from a physico-chemical and environmental point of view. Significant quantities of waste were collected or purchased. To preserve the initial moisture content of the products, they were stored in waterproof bags. They were then dried in an oven at 60°C until reaching a constant mass. This temperature preserves the initial mineralogical composition and the organic matter MO. A mechanical preparation phase follows, more precisely a sieving phase, in order to recover the part necessary for our use, which is stored in special waterproof laboratory bags. Following standardized methods and experimental plans, a first part is subject to physicochemical and mineralogical characterization. The remainder is intended to be used in manufacturing the final product, and the final product is the product of the manufacturing process.

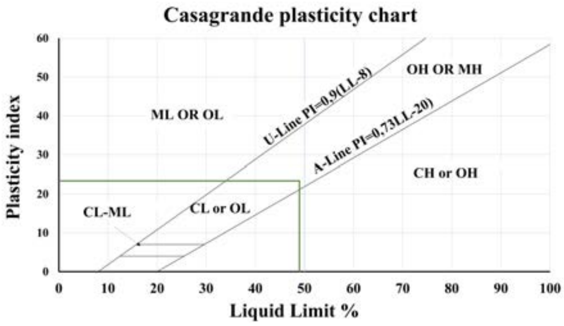


**Figure 10**. Plasticity chart.

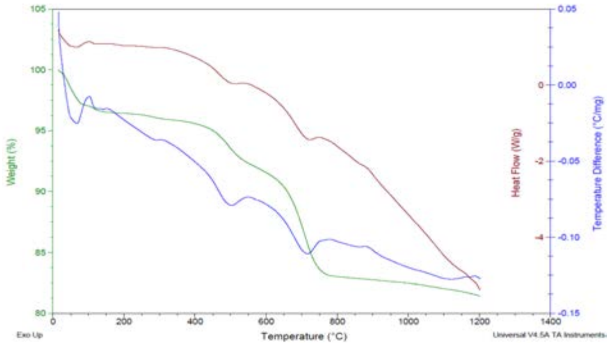


**Figure 11**. Thermogravimetric analysis of the samples: Mila clay.

This means that there are two different methods of sampling: for large quantities, it is done by quartering or manual fractionation, otherwise by means of dividers "samplers", devices separating into equal parts a quantity of material determined for the sample intended for the characterization of the product in the laboratory (68).

- The sand comes from the Sigues quarries located in Oum El Bouaghi province, in eastern Algeria. It was collected at a symbolic price, as it is intended for backfilling work, from sellers of building materials. It was sieved, keeping the fraction of 0.16–2 mm in diameter. 10% by weight of the clay was replaced with sand, as a common degreaser, to verify the effect of large calcite particles on the behaviour of the new bricks.
- The marble powder comes from carbonate metamorphic rocks (33) available in Algeria in huge quantities (40). Two grain sizes were used, fine marble powder (*FMP)* and very fine marble powder (*VFMP)* in order to study the effect of grain size on the bricks. These marble powders are commercially available from Bir Slam Activity Zone (Béjaia, Algeria). The data sheet lists the following composition: calcite and quartz, feldspar and mica impurities.
- Steel filings (*SF)* were taken from the wastes of blacksmith workshops (84). The material was sieved to keep only the particles between 1 and 2 mm.
- The wood sawdust (*WS)* was collected from woodworking workshops and used after sieving, keeping the dimensions between 5–10 mm.

*2.2.3.1. Chemical characteristics of additives*

The chemical composition of the different wastes was analysed by X-ray fluorescence (XRF). The results are presented in Table 6. The main constituents of the marble powder and sand are calcium (Ca) with 95.96 and 96.27% respectively, followed by aluminium (Al) with about 3.50% by weight and iron (Fe) with 0.30% and 0.13% by weight, while noting the presence of traces of (Si) in the sand. It is worth mentioning that although the presence of calcium (Ca) can have advantages during the firing process, such as the production of strong bricks, it can also have a negative effect on the porosity of the bricks (11). For the steel filings, the main component is (Fe) 81.31 %, followed by (Al) 9.70 %, (Ca) 4.92 %, (Si) 1.99 % and traces of (K) 1.99 %. Silicon and aluminium are two of the essential components of the clay bricks used in the walls. An iron content of 10% is also favourable for producing fired bricks. A higher content could lead to efflorescence, discolouration and black core formation when fired in an unfavourable atmosphere (85). In the wood sawdust, the components (Ca, Al, K, Si and Fe) are present in low percentages, 0.56%, 0.70%, 0.27%, 0.22% and 0.03% by weight respectively.

Furthermore, Table 6 shows that the waste contains small amounts of heavy metals such as (Cr, Mn, Ni, Cu, Zn, As, Rb and Pb). However, heavy metal phase transformation and volatilisation are strongly influenced by significant calcium oxide (CaO) concentrations. In addition, at high temperatures it is able to trigger the oxidation of Cr (III) to Cr (IV). This makes CaO beneficial in stabilising heavy metals. Most of the metals in these wastes would not be transformed into an unstable state. This means that the leaching of toxic metals is reduced (85).

*2.2.3.2. Physical characteristics, soluble salts and thermogravimetric analysis of additives*

Figure 12 shows the grain size distribution and Table 7 reports the grain size data together with the Methylene Blue Value. The results indicate that the grain size distribution of the marble powder is too tight for ≥70% of the fine portion for FMP and for ≥86% of the fine portion for VFMP. This difference can influence the behaviour of the specimens. The Sigues sand has a sand equivalent (SE) equal to 57.2%, therefore the fine fraction was eliminated, keeping only the portion above 0.16 and below 2 mm. The MBV of 0.75 (g/kg) confirms that it is insensitive to water (72). Table 7 shows the $CaCO_3$ content, which is 87.7% for Sigues sand and 90.4% for fine and very fine marble powder. This means that calcite is present in large quantities in the additions. The Sigues sand thermogravimetry curve (Figure 13) shows a single weight loss peak between 700 and 850 °C, of the order of 44.5%, corresponding to the decomposition of calcite (62, 78). Above the decarbonation temperature, calcite decomposes, releasing $CO_2$ and forming lime (CaO), which absorbs atmospheric moisture to form calcium hydroxide [Ca $(OH)_2$]. The latter slowly reacts with atmospheric carbon dioxide ($CO_2$) to crystallise once again in calcite (11).

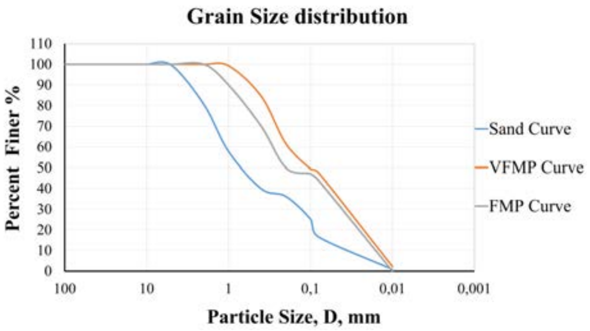


**Figure 12**. Grain size distribution of sand, FMP and VFMP.

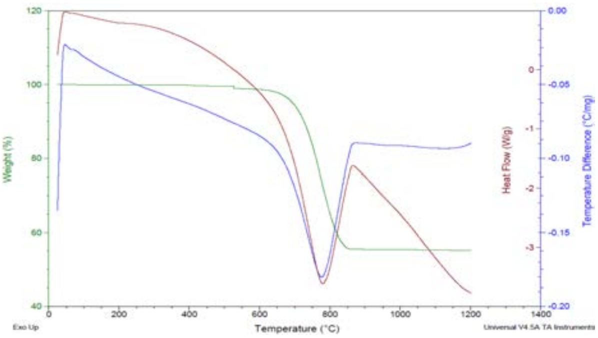


**Figure 13**. Thermogravimetric analysis of Sigues sand.

**Table 6**. Chemical characteristics of additives.

| Sample | Si % | Al % | S % | Cl % | K % | Ca % | Fe % | Mn % | Cr % | |
|---|---|---|---|---|---|---|---|---|---|---|
| FMP, VFMP | 0.00 | 3.70 | 0.00 | 0.00 | 0.00 | 95.96 | 0.30 | 0.02 | 0.00 | |
| Sand | 0.05 | 3.50 | 0.00 | 0.00 | 0.00 | 96.27 | 0.13 | 0.01 | 0.00 | |
| SF | 1.99 | 9.70 | 1.07 | 0.05 | 0.09 | 4.92 | 81.31 | 0.43 | 0.05 | |
| WS | 0.22 | 0.70 | 0.02 | 0.00 | 0.27 | 0.56 | 0.03 | 0.03 | 0.00 | |
| Sample | Co % | Ni % | Cu % | Zn % | As % | Rb % | Sr % | Zr % | Mo % | Pb % |
| FMP, VFMP | 0.00 | 0.03 | 0.01 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 |
| Sand | 0.00 | 0.00 | 0.01 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 |
| SF | 0.00 | 0.00 | 0.01 | 0.01 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| WS | 0.22 | 0.70 | 0.02 | 0.00 | 0.27 | 0.56 | 0.00 | 0.03 | 0.03 | 0.03 |

**Table 7**. Physico-chemical analysis of additives.

| Test | Sand | | | FMP | | | VFMP | | |
|---|---|---|---|---|---|---|---|---|---|
| **Sieve mesh size (mm)** | 0.080-0.315 | 0.315-1.25 | 1.25-5.00 | 0.080-0.315 | 0.315-1.25 | 1.25-5.00 | 0.080-0.315 | 0.315-1.25 | 1.25-5.00 |
| **Percent passing (%)** | 40 | 20 | 40 | 70 | 26 | 4 | 86 | 14 | 0 |
| **$CaCO_3$ %** | | 87.70 | | | 90.40 | | | 90.40 | |
| **Indissoluble %** | | 0.66 | | | 5.30 | | | 4.90 | |
| **Sand equivalent: SE %** | | | | | | | | **57.2 %: that means that a fairly clean sand** | (74) |
| **Methylene blue, sand: MB= (V1/M1) *1 (g/kg)** | | | | | | | | **0.75: the earth is insensitive to water** | (72) |

## 2.3. Brick samples manufacturing

This part deals with the process of bricks manufacturing. The clay was prepared by dehumidification, homogenization and grinding (Figure 14). Then it was sealed in plastic bags until further use, according to XP P94-202 (80). The manufacture of the control fired bricks (CMB) was carried out according to a traditional protocol with a percentage of water of 43% by weight of the clay as indicated in Table 8, a percentage that when added to the mixture leads to excellent plasticity, which will make it easier to handle the mixture, but causing an important shrinkage during firing (Figure 14-f). Non additivated reference bricks were prepared to evaluate the effect of using additives on the new sample properties.

For the production of the additivated clayey bricks, the minimum possible amount of water was added for kneading, a method tried by Laibi et al. (83) and Stazi et al. (86) to reduce the water absorbed by the mixture. The mixing was done with an electric mixer for 10 min and the clayey mixture was then left to age for a few days. During shaping, the wet clay was pressed into the mould by compacting using a rubber hammer. Unmoulding was carried out on abrasive paper to prevent early cracking during drying. Drying was carried out at ambient temperature until constant weight. Subsequently, the specimens remained in an electric dryer at 105 °C until constant weight. They were then fired in a muffle furnace (Nabertherm©) at 900 °C for 36 hours (Figure 15-e) (35). All brick specimens were then tested in the laboratory.

**Figure 14**. The steps of a traditional process to manufacture the series of non-additivated fired bricks: a) sieving; b) moistening; c) mixing; d-e) moulding; e) drying; f) firing.

**Figure 15**. Manufacturing of the additivated fired bricks after sieving, mixing of the additive and moistening steps: a) electric mixing; b) moulding by compacting using a rubber hammer; c) unmoulding carried out on abrasive paper; d) electric drying; e-f) firing.

Table 8 reports the mix proportion of the different fired bricks. It should be noted that the amount of water increases with the addition of 5% of sawdust, an additive that increases the demand for water to achieve good plasticity, the reason being the high absorption capacity of organic components (34).

## 3. RESULTS AND DISCUSSION

### 3.1. Fired Bricks colour

The fired bricks’ colour depends on the mineralogical and chemical composition of the raw materials of the additives (Figure 16) and on the firing temperature. The fired bricks with the addition of SF are darker, due to the great presence of iron in the form of $Fe_2O_3$ (31, 87). On the other hand, the addition of marble powder gives rise to a clear colour due to the presence of neoformation phases due to the reaction of CaO with silica (88, 89).

**Table 8**. The mix proportion of the different earthen mixtures (%).

| | Constituents | | | | per 100% weight of mixture | | |
|---|---|---|---|---|---|---|---|
| Mixtures | Mila clay | Sand | FMP | VFMP | SF | WS | Water |
| CMB | 100 | 00 | 00 | 00 | 00 | 00 | 43 |
| FMPB | 70 | 10 | 20 | 00 | 00 | 00 | 25 |
| VFMPB | 70 | 10 | 0 | 20 | 0 | 0 | 24 |
| SFB | 70 | 10 | 0 | 0 | 20 | 0 | 23 |
| WSB | 85 | 10 | 0 | 0 | 0 | 5 | 28 |

CMB = Control fired bricks; FMPB= fired bricks with fine marble powder; VFMPB= fired bricks with very fine marble powder; SFB= fired bricks with steel filings; WSB= fired bricks with sawdust.

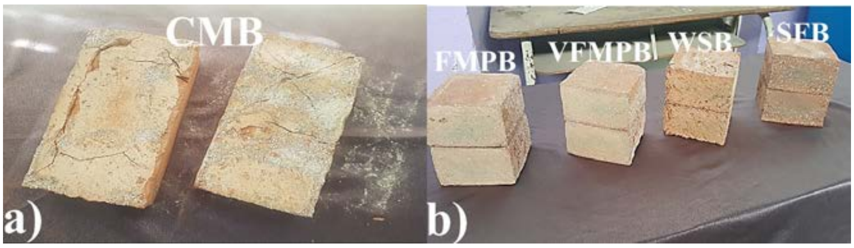


**Figure 16**. From right to left, a) fired bricks without additives (CMC); b) additivated fired bricks.

## 3.2. Physical characteristics

### *3.2.1. Bulk density*

Table 9 shows that bricks with the addition of FMP and VFMP have bulk densities of 1.60 g/cm$^3$ and 1.64 g/cm$^3$ respectively, which are lower with respect to the control fired bricks (CMB) (1.69 g/cm$^3$). A decrease which can be explained by the decomposition of calcite particles larger than 0.5 mm in size at high sintering temperature, causing the appearance of pores, cracks and a loss of weight linked to the release of $CO_2$, which reduces the density and increases the porosity. These results are consistent with previous researches (33, 88). The addition of 5% WS, determines a significant decrease of the bulk density (1.35 g/cm$^3$) due to the increase in porosity consequence of the combustion of the sawdust. The bricks added with SF show the highest density (1.87 g/cm$^3$) as a consequence of the high density of steel filings (31). Unlike the fired WS bricks, all the bricks are in accordance with the standards mentioned by Khitab et al. (22).

### *3.2.2. Firing shrinkage and weight loss*

The results show that the type of additive plays an important role in the shrinkage and weight loss of bricks, as displayed in Table 9. The shrinkage increases to reach a maximum of 11.3% for the reference brick CMB, which represents a 41.75% increase of weight loss, due to the great plasticity of the Mila clay. This promotes glassy phases, reducing porosity and increasing density and strength (87).

In addition, a moderate shrinkage decrease is observed for VFMPB and FMPB 6.9% and 6.4%, respectively, favoured by the good inter-particle cohesion in the liquid phase during the aging process, while a loss of mass remains evident, due to the decomposition of the calcite grains (78, 90). The appearance of secondary pores in the structure, due to the release of $CO_2$ during calcination of $CaCO_3$, is the main reason for the difference in size of FMP compared to VFMP (87). For SF, the moderate increase of 5.5% is mainly explained by the loss of water added during mixing. However, SF is also an inert component during the heat treatment, as evidenced by the lowest mass loss of 25.85% (46). The lowest shrinkage 4.7% is shown by the WS fired brick sample, since the wood does not shrink during firing. On the other hand, the weight loss is at its maximum 38.82 %, as wood burns under the effect of heat (87), giving rise to a lighter, more porous and less resistant material. All the shrinkage results conform to ASTM standards (29, 51).

**Table 9**. Physical and mechanical properties of the fired bricks.

| Sample | WSB | SFB | FMPB | VFMPB | CMB | SFB |
|---|---|---|---|---|---|---|
| $\gamma_s$ (g/cm$^3$) | 1,35 ±0.03 | 1,87±0.02 | 1.60 ±0.01 | 1.64 ±0.03 | 1.69 ±0.02 | 1.74±0.02 |
| R (%) | 4.7±0.4 | 5.5±0.5 | 6.4±0.7 | 6.9±0.4 | 11.3±0.4 | / |
| $W_L$ (%) | 38.82 ±0.14 | 25.85±0.50 | 36.11 ±0.40 | 32.65 ±0.23 | 41.75 ±0.30 | / |
| (AW)(kg/m$^2$.min) | 8.72 ±0.45 | 1.12 ±0.80 | 0.90 ±0.70 | 0.79 ±0.60 | 0.72 ±0.90 | 8.79 ±0.40 |
| P (%) | 44.6±0.1 | 33.5±0.1 | 30.3±0.0 | 29.3±0.0 | 27.2±0.1 | 31.0±0.1 |

Note: (WSB= fired bricks with sawdust; SFB= fired bricks with steel filings; FMPB= fired bricks with fine marble powder; VFMPB= fired bricks with very fine marble powder; CMB = Control fired bricks; SFB= Skikda fired bricks; γs (g/cm$^3$) = bulk density; R% = Firing shrinkage; WL (%) = Weight loss; AW (kg/m$^2$.min) = Water absorption; P (%) = total open porosity).

*3.2.3. Water accessible porosity and capillary water absorption*

The bricks without additives show a water accessible porosity and water absorption of 27.2% and 0.72 (kg/m$^2$.min) respectively (Table 9). The VFM fired bricks show a high increase in porosity and water absorption (30.2% and 0.90 (kg/m$^2$.min) respectively) due to $CaCO_3$ decomposition during firing, which turns into calcium oxide, causing an increase in porosity linked to the release of $CO_2$. In contrast a moderate increase in porosity and water absorption is observed for VFMP fired bricks (29.3%, 0.79 (kg/m$^2$.min) respectively). This moderate increase in porosity (with respect to the bricks added with VFM) can be explained by the greater ease of small CaO granules to react with silica. Also, the addition of SF increases the porosity and absorption (33.5%, 1.12 (kg/m$^2$.min)), due to the high coefficient of thermal expansion of the metal compared to the fired brick matrix, which causes stresses in the material (31, 46, 84).

The addition of 5% WS gives rise to an absorption of 8.72 kg/m$^2$.min and a porosity of 44.6% (20, 91). It should also be mentioned that the amount of sand added to the WC and SF fired bricks further increases the percentage of porosity. Moderate porosity of bricks improves their thermal insulation properties by maintaining pleasant temperatures inside the building, which also reduces energy consumption. Conversely, very high porosity can pose problems (87). As a main conclusion, despite the increase in porosity and water absorption, the fired bricks added with SF, VFMP and FMP comply with NF EN 771-1+A1/CN standards (57), unlike those with WS, which exceed the water absorption and porosity allowed (22, 91).

*3.2.4. Compressive strength*

Table 10 and Figure 17 show the compressive strength of the fired bricks with the different additives. The results show a slight increase in resistance 4.6 MPa for the control fired bricks (CMB) with respect to the Skikda old fired bricks 4.3 MPa. The fired bricks with VFMP show the best mechanical properties, with a compressive strength of 11.7 MPa. As observed by Thalmaier et al. (89), Darweesh (91) and Muñoz et al. (92), the decomposition of fine marble grains gives rise to fine pores as well as to the formation of calcium silicates and aluminates, thus favouring the hardening of the fired brick during the firing process. This is followed by fired bricks with the addition of FMP and SF, with resistances equal to 9.7 MPa and 9.6 MPa, respectively. As regards FMP, Achik et al. (62) and Sutcu et al. (82) state that the addition of coarse calcite can be dangerous because the coarse calcium oxide particles produced during firing will not react with silica but will hydrate and carbonate in the masonry, generating stress that worsens the mechanical characteristics of the fired bricks. Researches such as that of Shehbaz et al. (88), state that coarse marble powder larger than 0.5 mm is detrimental to the development of good mechanical properties due to greater development of porosity. This phenomenon is recognized as the "lime blowing" phenomenon (93, 94). As for the addition of SF, the increase in compressive strength can be explained by the inert behaviour during firing and by the increase in bulk density. This point has been addressed by previous studies such as (46, 94). The addition of WS is detrimental for the mechanical properties because of the great increase in porosity due to the combustion of the wood particles when firing (62, 95). According to EN 771-1/CN and ASTM C-67, VFMP, FMP and SF can be considered appropriate for exterior and interior walls (52, 96, 97), unlike WS (34, 82).

*3.2.5. Tensile strength*

Table 11 and Figure 18 show the tensile strength of the fired bricks as a function of the additions. In contrast to the compressive strength, the Mila control fired brick shows a lower tensile strength 1.5 MPa compared to that of Skikda old fired brick 1.7 MPa. This decrease is due to the cracking of the control fired bricks CMB, given the high clay content.

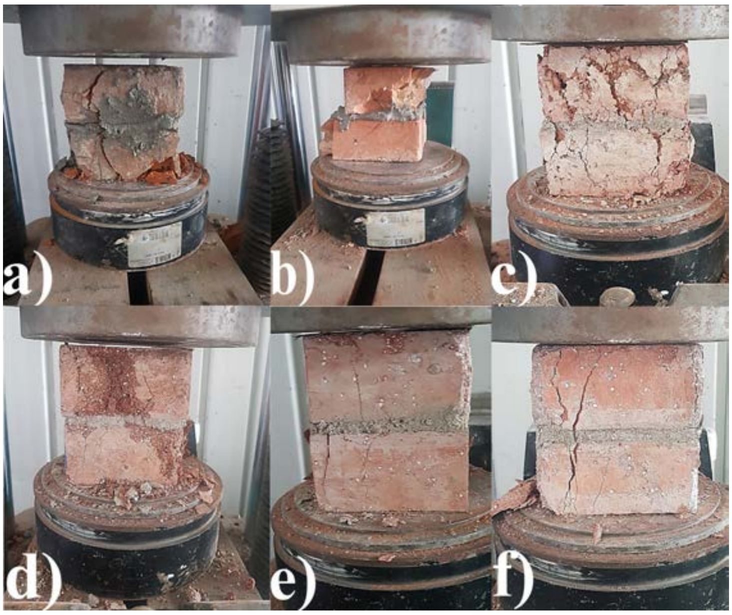


**Figure 17**. Uniaxial compression test on representative specimens: (a) SOFB; (b) CMB; (c) WSB; (d) SFB; (e) VFMPB; (f) FMPB.

**Table 10**. Uniaxial compressive test (MPa).

| | Compressive strength (MPa) |
|---|---|
| SOFB | 4.3±0.4 |
| CMB | 4.6±0.5 |
| WSB | 2.2±0.2 |
| SFB | 9.6±0.6 |
| FMPB | 9.7±0.5 |
| VFMPB | 11.7±0.4 |

**Table 11**. Indirect tensile test (MPa).

| | Tensile strength (MPa) |
|---|---|
| SOFB | 1.7±0.3 |
| CMB | 1.5±0.4 |
| WSB | 0.6±0.2 |
| SFB | 1.9±0.2 |
| VFMPB | 2.7±0.4 |
| FMPB | 2.6±0.3 |

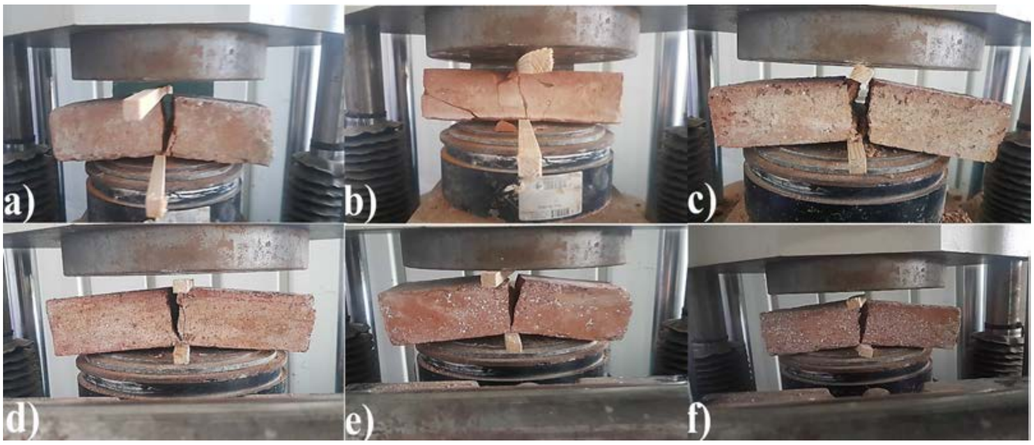


**Figure 18**. Indirect tensile test on a representative specimen: a) SOFB; b) CMB; c) WSB; d) SFB; e) VFMPB; f) FMPB.

The most satisfactory results are for VFMPB 2.7 MPa. Ahmad (88) and Elert et al. (94) showed that such an increase can be explained by the presence of a significant amount of fine marble grains which gives rise to neoformation minerals (as previously reported), reducing the weak zones of the sintered fired bricks and improving tensile strength. With regard to the FMP fired bricks, the resistance obtained is less than 2.6 MPa, results that show the impact of the decomposition of the coarse calcite under the effect of heat, generating stresses which impact the resistance to tension of the fired bricks (41, 78, 94). On the other hand, the addition of SF gives a resistance of 1.6 MPa, lower compared to bricks with FMP, mainly due to the increase in porosity. In fact, steel filings reduce the plasticity and bonding capacity between particles. In addition, the alkaline medium hinder the transformation of heavy metals into a stable mineral phase at high temperatures thus decreasing the tensile strength, as reported by other scholars such as Dai et al. (36, 46). The lowest resistance is shown by the fired brick with the addition of WS, which reduces the connection between the particles and increases porosity during firing. These results confirm that the tensile strength is affected by the increased porosity, which in turn depends on the mass loss and microstructural properties of the fired bricks. It is worth mentioning that all the bricks showed the appearance of calcite grains, but to different degrees. This means that the cause of this reaction is probably particles of sand with a diameter >0.5 mm.

### 3.3. Conservation state threats

*3.3.1. Amount of calcite in Mila fired bricks and all fixed fired bricks*

Table 12 shows the amount of $CaCO_3$ present in the fired bricks after firing. 0.81% is the percentage found in CMB, 2.84% for SFB, 3.45% for WSB, 4.06 VFMPB and 7.52 for FMPB. This calcite is the result of the hydration and carbonation of CaO produced during firing. It is either primary and is already present in the raw material, or it is obtained by the addition of sand and marble powder.

**Table 12**. $CaCO_3$ content of the fired bricks.

| Sample designation | FMPB | VFMPB | SFB | WSB | CMB |
|---|---|---|---|---|---|
| $CaCO_3$ % | 7.52 | 4.06 | 2.84 | 3.45 | 0.81 |

*3.3.2. Comparison of SEM micrographs of fired bricks at magnification of 20 µm*

The fired bricks additivated with different waste materials have been evaluated and compared with the Skikda old fired bricks. (Figure 19-a) shows that the Skikda old fired bricks have a relatively compact, dense and smooth texture with a tight porosity. The Mila control brick (Figure 19-b) shows an open texture consisting of sheets with a random orientation that leave space to pores that are both open and unconnected (98). Calcite crystals are also visible, formed as a result of the decomposition of the original calcite (35, 99). The VFMP fired bricks (Figure19-c) show a more homogeneous texture, with the presence of a remarkable microporosity and small calcite grains as well as large ones. There appear to be a few potential reactions when calcium carbonate decomposes, the new calcium silicate phases are very fine. Moreover, the presence of small pores resulting from the decomposition of fine calcite grains can be observed together with microcracks produced during expansion caused by hydration and carbonation of CaO originated by large calcite grains (98, 100). (Figure 19-d) shows the FMPB texture, where large pores can be observed, phenomenon linked to the decomposition of marble and carbonate sand powder with a diameter greater than 0.5 mm (11, 35). Moreover, large calcite grains are present, result of the CaO hydration and carbonation. This process can give rise to microcracking due to crystallization in a confined space (100, 101).

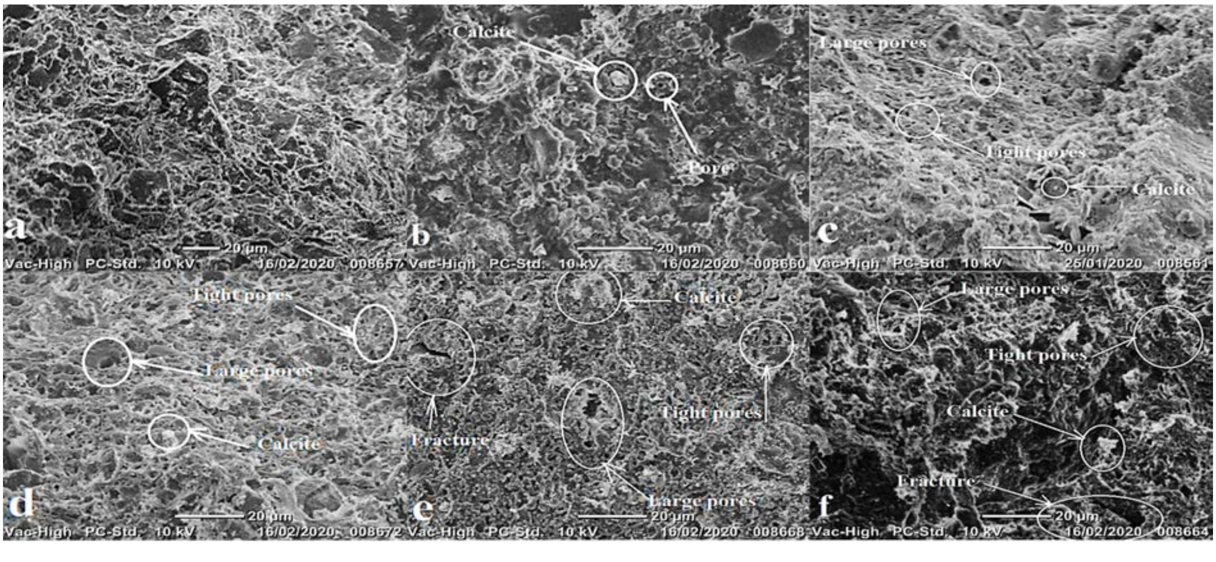


**Figure 19**. Micrograph obtained by SEM of the bricks sintered at 900 °C:
a) SOFB; b) MCB; c) VFMPB; d) FMPB; e) WSB; f) SFB; at magnification of 20 µm.

(Figure 19-e) shows the sample with 5% WS, which appears rough and uneven (98). The images of the crumbling surface explain the fragility of the fired bricks. The heterogeneous organization of grains, which reveal cracks, and the appearance of loose particles and micro-cracks are clear signs of alteration. The calcite particles are very large, due to the carbonation of the CaO originated from the coarse calcite particles found in the earth and sand. Also, a large porosity is seen as a result of the combustion of wood.

(Figure 19-f) represents the matrix of SFB, reflecting a less dense matrix and a less heterogeneous organization of the grains. Fractures and micro-fissures appear, a clear sign of physical weathering, as the steel filings reduce the plasticity and the bonding capacity between the particles, as confirmed by Daï et al. (36, 98). There are also large pores which are due to the decomposition of the carbonate sand particles during firing (46). At 900 °C the morphology and signs of vitrification are only partially observed.

### 3.4. Environmental aspect

The environmental aspect of the fired bricks was analysed by means of the leaching test. The leaching test was performed using the USEPA 1311 method. Table 13 shows the cumulated leach results. The heavy metals selected for investigation are (Cu), (Pb), (Zn), (Mn), (Ni), (Cr), (Fe), (As) and (Cd). The maximum concentrations of chromium (Cr), manganese (Mn), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As), lead (Pb), silver (Ag) were: 2,92 ppm, 3,94 ppm, 0,33 ppm, 1,36 ppm, 1,69 ppm, 0,05 ppm, 0,24 ppm, 0,91 ppm respectively for the bricks MCB, FMPB, VFMPB, SFB, WSB. We found that the cumulative leaching of all heavy metals is insignificant because it is well below maximum leaching limits established by United States Environmental Protection Agency (US EPA, 1996) (85).

**Table 13**. Toxicity Characterisation Leachate (TCLP) method (US-EPA 1311) (53, 54, 85, 105).

| Heavy Metals | Concentration Limit (mg/l) (USEPA, 1996) | Concentration (mg/l) | | | | |
|---|---|---|---|---|---|---|
| | | FMPB(ppm) | VFMPB(ppm) | WSB (ppm) | SFB(ppm) | CMB (ppm) |
| Chromium (Cr) | 20 | 0.44 | 0.38 | 0.26 | 0.28 | 0.92 |
| Manganese (Mn) | 260 | 1.62 | 1.82 | 1.18 | 3.94 | 1.52 |
| Nickel (Ni) | 8 | 0.28 | 0.24 | 0.20 | 0.33 | 0.19 |
| Copper (Cu) | 2 | 0.23 | 0.25 | 0.18 | 1.36 | 0.18 |
| Zinc (Zn) | 1200 | 1.69 | 0.85 | 0.65 | 0.61 | 0.43 |
| Arsenic (As) | 2.8 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 |
| Cadmium (Cd) | 0.8 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| Lead (Pb) | 5 | 0.03 | 0.04 | 0.03 | 0.01 | 0.24 |
| Silver (Ag) | 5 | 0.19 | 0.09 | 0.09 | 0.10 | 0.08 |

Leaching of (Cr), (As) and (Pb) from fired bricks, particularly those containing sawdust, showed reduced leaching. This suggests that the leaching capacity of some of the heavy metals could be reduced by the incorporation of biosolids into the fired bricks (102). The results also show that the levels of (Cr), and (Pb) in the control bricks are higher than the levels in the stabilised bricks as a consequence of the oxidation of heavy metals during the high-temperature firing process. Alternatively, the high silica content of the clay could improve the bonding between the particles in the bricks reducing the leaching of the heavy metals present in the raw material (103, 104). In addition, iron oxide may also contribute to the stability and formation of the network, including leaching. On the other hand (as mentioned above), studies have shown that the evaporation phase of heavy metals depends on the presence of high concentrations of calcium oxide (CaO). This is because high heat induces the oxidation of Cr (III) to Cr (IV), making CaO beneficial by stabilising heavy metals, and generally most of the metals contained in these wastes which would not be converted to unstable states. Therefore, this stabilisation decreases leaching of toxic metals (85).

This study shows a positive result with respect to possible heavy metal leaching when using waste as a partial solution to replace clay earth. This suggests that bricks incorporating waste could be used as a building material without treatment and without environmental impact. This will also help to reduce the pollution caused by used wood, marble and steel.

## 4. CONCLUSIONS

Through a series of tests carried out to characterize the old Skikda fired bricks and evaluate the behaviour of the new-fired bricks, the following conclusions can be drawn:

- The Skikda old fired bricks show mechanical strength under the required standard. This can be due to the degradation they have undergone over time. The mineralogical analysis revealed the presence of mullite and hematite, minerals of neoformation suggesting a firing temperature between 800 and 900 °C. This kind of minerals are formed in earths with a low calcium carbonate content;

- Mila clay is considered suitable for fired bricks production. However, its swelling characteristics favouring very high shrinkage require appropriate correction of the grain size;
- The fired bricks with 20% VFMP show the best compressive strength. The decomposition of the fine marble grains, gives rise to fine pores and to calcium silicate minerals, ensuring good cohesion;
- In contrast, in the case of FMP fired bricks, a decrease in strength and an increase in porosity have occurred, due to the decomposition of the coarse calcite grains and to the stress caused by the hydration and carbonation of the CaO;
- 20% of steel filings (SF) improves the mechanical behaviour of the bricks, reduces the plasticity of the mixture and shrinkage, and increases the density. Nevertheless, an increase in porosity and water absorption is also observed;
- Sawdust (WS) burns during firing, leaving large pores and reducing the number of connections between clay grains. This increases the absorption capacity and decreases the strength. On the other hand, it considerably lightens the material;
- The Sigues quarry sand is not suitable as a degreaser because of the large grain size of the calcite, it must be finely ground for it to be suitable;
- As the main conclusion of this work, steel filings and very fine marble powder seem to be satisfactory and cheap waste for the correction of the clayey raw material to produce suitable fired bricks to be used in rehabilitation interventions. Future researches should determine the appropriate percentage of additives and an ideal grain size to avoid the appearance of the lime blowing phenomenon. Nevertheless, to minimize damage, fine grinding of the clay and additives is recommended, together with the immediate immersion of the fired bricks in water after firing so that the grains of CaO could be solubilized and carbonation does not occur inside them.
- Furthermore, the leached metal concentrations for the different bricks comply with USEPA. Therefore, the four waste materials have good potential for appropriate, sustainable and safe use as raw materials in bricks production.
- The acquired results can be part of a database of industrial waste, which can be used in the mass production of fired bricks thus allowing professionals in the field to select the right earth and additives capable of producing a good durability.

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**Declaration of Competing Interest**

The authors of this article declare that they have no financial, professional or personal conflicts of interests that could have inappropriately influenced this work.

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