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Towards the Use of Yellow Clay in Fired Bricks

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Abstract

This chapter deals with the study of the possibility of using yellow clay - which was only used in pottery so far- in the civil engineering field as building materials, especially in the field of fired bricks. With the aim to improve the technological properties of yellow clay based bricks, two wastes were used as secondary raw materials. The first one is a mineral waste - pyrrhotite ash - this waste was neither characterized nor valued before by any other author. While the second waste is an organic waste - cedar sawdust - which is from the artisanal sector. Clay bricks containing yellow clay and different content of wastes were prepared and tested to evaluate their technological properties: water absorption, bulk density, porosity and mechanical strength... The test results indicate that the addition of wastes to clay bricks improves their technological properties and highlights the possibility of wastes reuse in a safe and sustainable way.

Keywords: clay, bricks, mineral waste, organic waste, mechanical strength, clay bricks, waste, pyrrhotite ash, recycling

1. Introduction

Recently, the study of the reuse of industrial solid waste in the fields of construction materials (fired bricks, tiles, etc.), pavement materials and concrete has received considerable attention across the world. The recovery of wastes is used to develop environmentally friendly technologies, to reduce negative impact on the environment and landfill waste in large storage and disposal areas, and to reduce production costs for new products. However, the recovery of wastes depends on their chemical composition, their microstructure and their physical and hydro-dynamic properties. For example, industrial waste containing iron oxide, silicon oxide, aluminum oxide - in the form of major oxides with various contents - such as fly ash (with major oxides: SiO_2 , Al_2O_3 , Fe_2O_3), pyrite ash (major oxide: Fe_2O_3), red mud (major oxides: Fe_2O_3 , TiO_2 , Al_2O_3 , SiO_2), as well as biomass ash, were studied and reused in various fields. There are many applications for these wastes:

- Fly ash is mainly used in the production of concrete, road bedrock materials, cement clinkers and geopolymer concrete [1, 2];
- The red mud is used to produce ceramic blocks which store heat [3, 4]. It is also used in the plastics industry and for the production of pigments and bricks for the construction industry, road construction and agriculture [5];
- Pyrite ash is used to produce high density materials for heat storage materials and road construction materials [6];
- Biomass ash such as bagasse ash from sugar cane, rice husk ash and wood ash [7–9] for the production of bricks based clay.

On the other hand, the reuse of wastes in the field of terracotta is not the only reason to conduct research on the addition of certain solid residues in a clay matrix, even though it was the ultimate goal of this research. Other reasons can be taken into consideration. In fact, waste can:

- Economize energy in the manufacturing process by increasing the temperature at certain stages of the firing process in a tunnel kiln. Their higher calorific values cause self-combustion within the clay matrix so that a minimum of energy is required to firing the bricks;
- Reduce water requirements while improving the plasticity of the mixture;
- Improve - according to its chemical and mineralogical composition - certain technological properties of clay bricks.

Therefore, the reuse of wastes as additives in the ceramics sector has more reason to be than recycling. So, various research has focused on improving the performance of clay bricks. Some authors have studied the effect of wastes on manufacturing processes - and more specifically the firing process - and others have estimated the amount of waste that can be added to meet masonry standards. It consists of the evaluation of the technological properties of bricks versus the nature of the waste and the rate of its incorporation into the clay matrix.

The literature shows that the addition of waste glass improves the compressive strength, water absorption and porosity rates of bricks. Also, this waste increases the shrinkage of bricks [10, 11].

The addition of other additives such as sawdust and marble residue resulted in bricks with good compressive strength, especially for 15–20 wt.% of marble powder content [12]. The water absorption of these bricks was very high to be used in the field of civil engineering [13].

Another example of organic waste concerns the addition of biomass ash such as sugar cane bagasse and rice husk ash to clay bricks. These wastes reduce mechanical strength and increase the ability of bricks to absorb water [7, 8]. These two types of bricks have interesting thermal insulation properties and are also lighter which is an advantage in terms of transportation and use in the areas affected by the earthquake [8, 14].

The performance of clay-based bricks containing various amounts of rice husk ash or wood ash was also evaluated. The study has shown that bricks containing up to 10 wt.% of the rice husk ash and those containing 30 wt.% of the wood ash respect the standard requirements of clay masonry units [15]. Other work on the same waste has shown that 20 wt.% of the wood ash can be added to the ceramic

matrix as a natural, economical and environmentally friendly pigment, thus allowing the lightening of bricks [16]. Another study has shown that adding sawdust to clay bricks improves porosity and results in lightweight bricks [17].

Among the ash waste category, there is another industrial waste, rich in hematite Fe_2O_3 , which is the pyrrhotite ash. Few studies were carried out for this waste to explore areas of its recovery in industry [18–22]. It was generated between 1964 and 1982 [1] by the sulfuric acid manufacturing process from the combustion of pyrrhotite ore extracted from the Kettara mine in Marrakech (Morocco). Pyrrhotite ash is currently stored in large quantities in a large open space in the southwest of Morocco.

The present work is a contribution to evaluate the effect of the addition of two types of wastes on the technological properties of fired bricks-based yellow clay. This clay, whose main components are silica, calcium carbonate and kaolinite, was only used in pottery so far. The two wastes used are the pyrrhotite ash, which is a mineral waste, and the cedar sawdust, which is an organic waste from the artisanal sector.

2. Method and materials

The approach followed throughout this study is presented in **Figure 1**. The chemical, physical, mineralogical, environmental, thermal and mechanical characterization were carried out. Many analytical techniques were used, namely: X-ray diffraction (XRD), X-ray fluorescence (FRX), Fourier transform infrared (IR) spectroscopy, reservoir, Inductively Coupled Plasma Spectrometry (ICP),

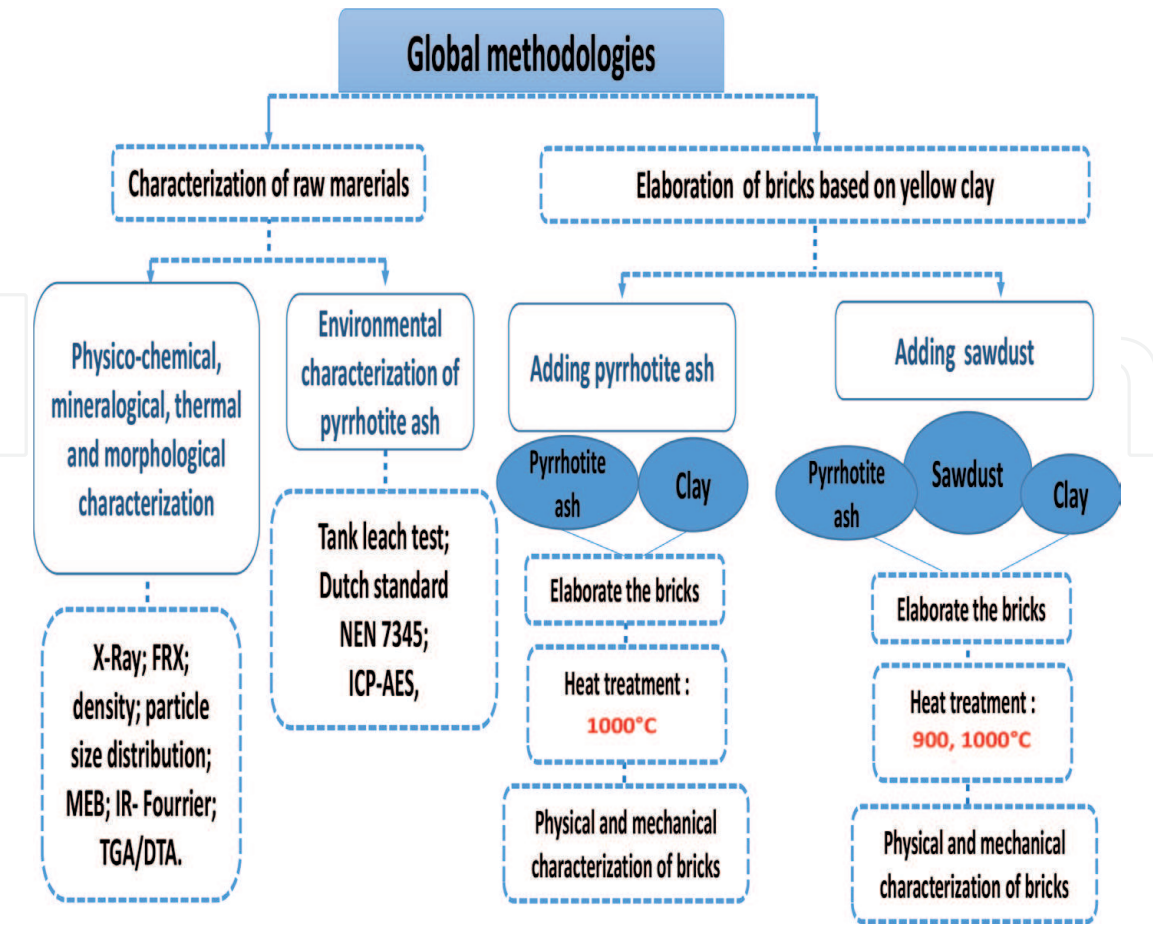


Figure 1.
Global methodologies used to carry out this study.

Thermogravimetric Analysis (TGA), Differential Thermal Analysis (DTA), Hydrogen potential (pH), Scanning Electron Microscopy (SEM), distribution particle size, bulk density, apparent porosity, shrinkage, weight loss, water absorption, three-point flexural strength and compressive strength.

Three raw materials were used, namely:

- The yellow clay extracted from the region of Fez city, and used as a matrix to make bricks;
- Pyrrhotite ash produced during the manufacture of sulfuric acid from pyrrhotite ore extracted from the Kettara mine which is located in south west of Morocco. The sample used in this study was retrieved from the depth of 0.5 m. It is a mineral waste that was used as an additive to improve the mechanical properties of the produced bricks;
- Sawdust from cedar wood, organic waste which is a waste of the artisanal sector of the old medina of Fez. It was incorporated into the ceramic body to adjust the physical properties of bricks.

The characterization of the raw materials concerns the identification of:

- The particle size distribution of pyrrhotite ash was carried out using the Sifting machine DIGITAL FI-FTL0150 & FIFTL0200. To achieve this analysis, sample was grinded with a wooden roller. The median diameter of particles was determined using the SediGraph 5100 instrument;
- The mineralogical phases using X-ray diffraction using Siemens D500 Diffractometer. The XRD patterns were performed in the range of 2θ between 2° and 60° with operating conditions of 40 kV and 50 mA;
- The functional groups using infrared Fourier transform spectroscopy (JASCO 4000 Fourier Transform Spectrometer) in wave number range of $4000\text{--}400\text{ cm}^{-1}$;
- The major and minor elements by means of X-ray fluorescence;
- The thermal behavior through thermogravimetric and differential thermal analysis (TGA - DTA) was performed with a TA Instruments balance model STA-1640. It allows obtaining simultaneous DTA and TGA diagrams under similar experimental conditions. Experiments were performed under N₂ flow, from $25\text{--}1000^\circ\text{C}$ at a heating rate of 5°C /min .

The **Figure 2** shows the steps of making bricks and the characterization of the fired bricks with different contents of pyrrhotite ash and sawdust.

- The morphological characteristics were determined by scanning electron microscopy (SEM) using a SEMHitch S-2500 microscope operating with an acceleration voltage of 16kv.
- The porosity and bulk density measured by the mercury porosimeter using Micromeritics AutoPore IV 9500 Series, and interpreted according to Swiss standards SIA 266 and ASTM C62 standards for porosity, and NF P 94–093 standard for density.

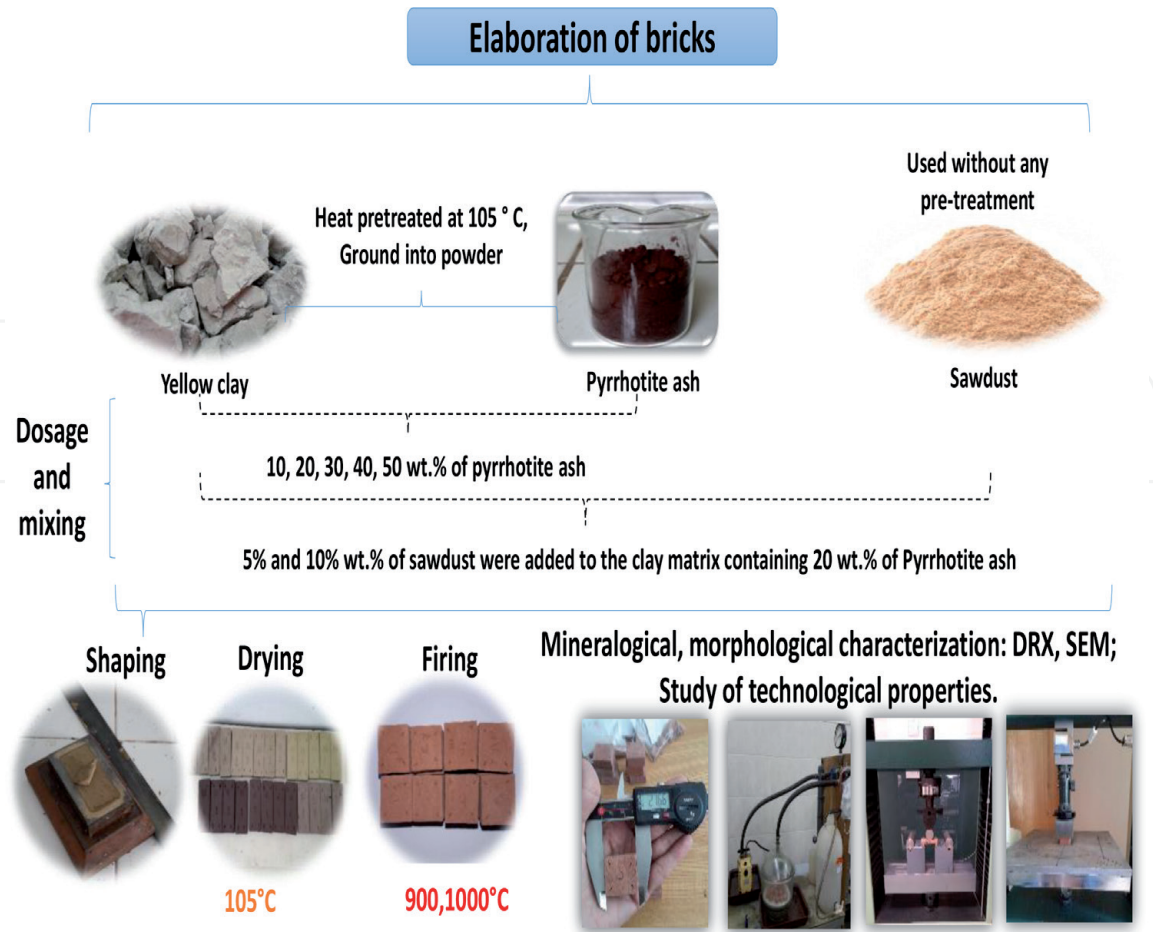


Figure 2.
The procedure adopted to carry out this work from the raw materials to the final bricks.

- The flexural properties have been carried out according to standard EN 771–1.
- The water absorption in accordance with ASTM C62.
- The shrinkage and weight loss according to ASTM and CNS 382.R2002 (Chinese National Standard).

3. In-depth study of the clay matrix

3.1 Summary of the chemical and physical properties of the yellow clay

The **Figure 3** shows the percentages of metal oxides obtained by XRF analysis. The **Table 1** shows the physicochemical characteristics of the clay with the main remarks drawn from this analysis.

The X-ray fluorescence analysis of yellow clay (**Figure 3**) shows the presence of several chemical elements [22]. According to the literature [23–25] this clay seems to be a calcareous clay which can be used in the production of low refractory building bricks.

As shown in **Table 2**, analysis by infrared spectroscopy of the clay reveals the presence of characteristic bands of silica, calcium carbonate and kaolinite. These results were confirmed by the X-ray analyzes which show the presence of crystalline phases of silica, calcite and kaolinite [22].

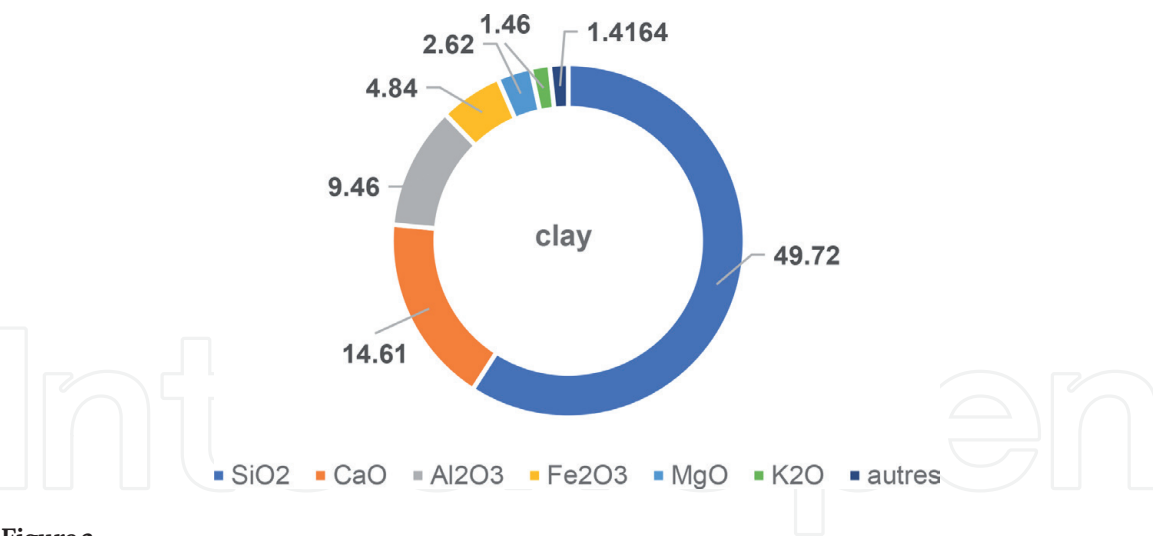


Figure 3.
X-ray fluorescence of clay raw.

Clay used for construction	Yellow clay	Interpretation
50 < SiO ₂ wt.% < 60	49,72	Can be used for the production of bricks
CaO wt.% > 6%	14,61	Limestone clay
ΣFeO, CaO, MgO > 9%	4,84 + 14,61 + 2,62 = 22.07% > 9%	Low refractory material

Table 1.
The main chemical properties of yellow clay.

	Yellow clay
Predominant crystalline phase (XRD analysis)	Silica, calcite, kaolinite, dolomite and albite
Characteristics bands (infrared spectroscopy analysis)	Silica, calcium carbonate and kaolinite

Table 2.
The main characteristics of yellow clay.

3.2 Thermal study of clay bricks

The chemical analysis of the clay highlights the possibility of using it to develop terracotta bricks. Given that this clay has never been used as a clay bricks, and in order to understand its behavior at different temperatures to establish a firing program, a thermal study was realized.

3.2.1 Thermal expansion test

This measurement was studied by the DIL 402 Expedis dilatometer with a heating rate of 3°C/min. The test was carried out on bricks made of clay with percentages of pyrrhotite ash: 0 wt.%, 30 wt.% and 60 wt.%. The expansion curves obtained are shown in **Figure 4**.

- L0: Length of the brick before thermal expansion;
- L: Length of the brick during thermal expansion;
- dL / L0: the thermal expansion factor.

From the results of the thermal expansion illustrated in **Figure 4**, and DTA / TGA detailed in a previous publication [22], two aspects are noted:

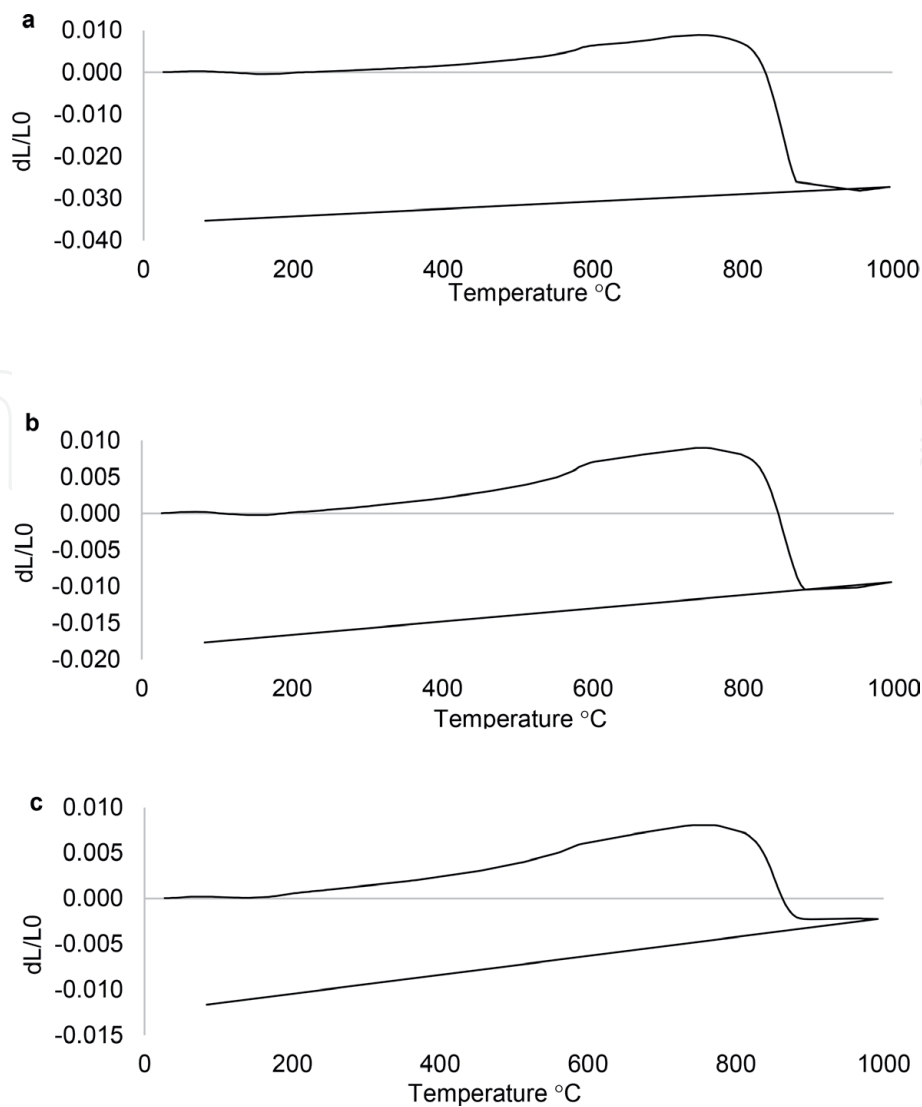


Figure 4.
Expansion curves for clay-based bricks with different pyrrhotite ash content: (a) 0 wt.%; (b) 30 wt.% and (c) 60 wt.%.

- Firstly, the expansion profile gives an idea of the shrinkage of bricks produced depending on the pyrrhotite ash content. **Figure 5** shows that the addition of ash decreases the dL/L_0 factor, which makes bricks able to keep their original shapes and improves their physical properties. Indeed, the decrease in dimensional variations reduces the risk of cracking that can affect the physical and mechanical properties of the ceramic body, namely porosity, density, resistance to mechanical force, etc.
- Secondly, dilatometric analysis allows to define the firing program, which is essential to approach the elaboration of any ceramic material, giving information on the temperature ranges where the material expands continuously without undergoing any deformation.

In this case, thermal expansion analysis (**Figure 4**) shows that all samples expand continuously without any deformation detected up to 750°C.

3.2.2 Brick heat treatment program

Based on the DTA/TGA analysis [22] and the dilatometry test, four temperature ranges can be identified. These areas are illustrated in **Table 3**. The identification of

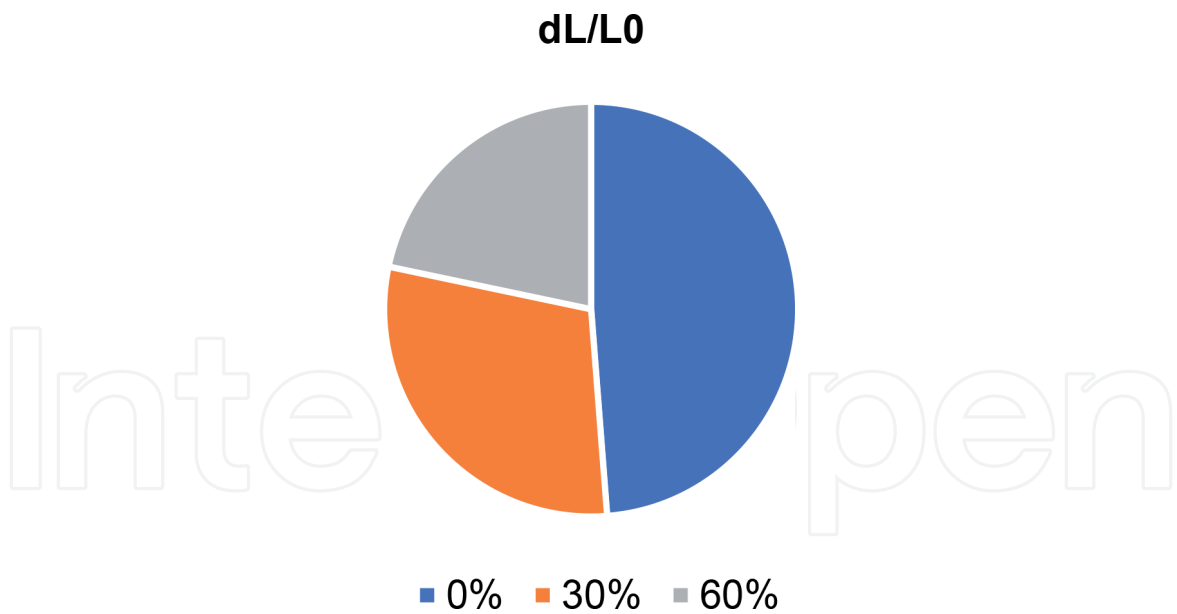


Figure 5.
Brick expansion profiles for bricks with 0, 30 and 60 wt.% pyrrhotite ash.

Temperature range °C	Heating rate °C/min	Observations
20–200	8	Moisture removal: hygroscopic water (100 °C);
200–350	2	Slow speed so that the combustion of the organic matter takes place without the appearance of micro cracks which can be caused by the release of gases from combustion; (260–347°C);
350–750	8	Fast speed where the material expands continuously;
750–1000	2	Slow speed for better decomposition of carbonates (813.31° C);
1000–20	1	Slow speed to avoid any structural deformation.

Table 3.
Justification of the adopted firing program.

these areas allowed to establish the most suitable heat treatment program for firing bricks made from yellow clay and pyrrhotite ash. This firing program highlights the treatment temperature as a function of time as well as the heating rate. **Figure 6** shows the established program.

After studying the possibility of making terracotta bricks-based on yellow clay from Fez, new elements are sought to be used as additives in order to improve the technological properties of bricks. Pyrrhotite ash was chosen to this end. It is a mineral waste that was studied and valorized for the first time in our previous work [19, 21]. The physicochemical characterization of the pyrrhotite ash showed that it is a mineral waste rich in hematite, silica and alumina. It is also weakly hygroscopic and exhibits a low loss on ignition (3.4%) with a density around of 4.33 g/cm³.

Morphological analysis by the scanning microscope shows that the pyrrhotite ash particles have a spherical shape with a relatively smooth surface. Obviously, the particles having a spherical shape and a smooth surface retain a small quantity of water, which confers to the pyrrhotite ash a weakly hygroscopic character.

The particle size of the pyrrhotite ash is continuous and contains grain fractions having a diameter between 1 µm and 125 µm. It is well known that mixing different fractions generally led to a compact material because the relatively small particles can get lodged in the interstices between the larger ones. So the material

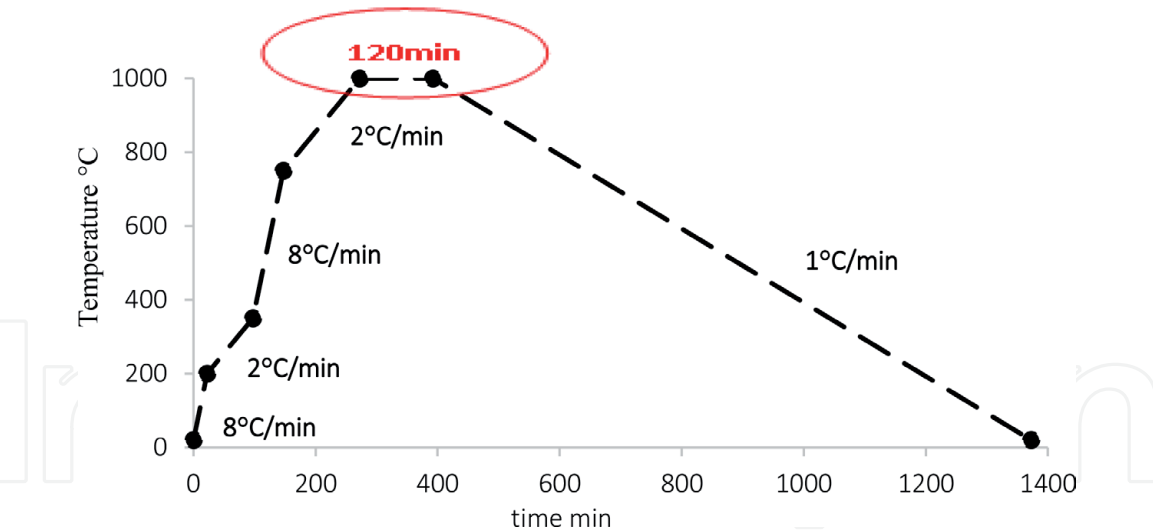


Figure 6.
Clay firing program.

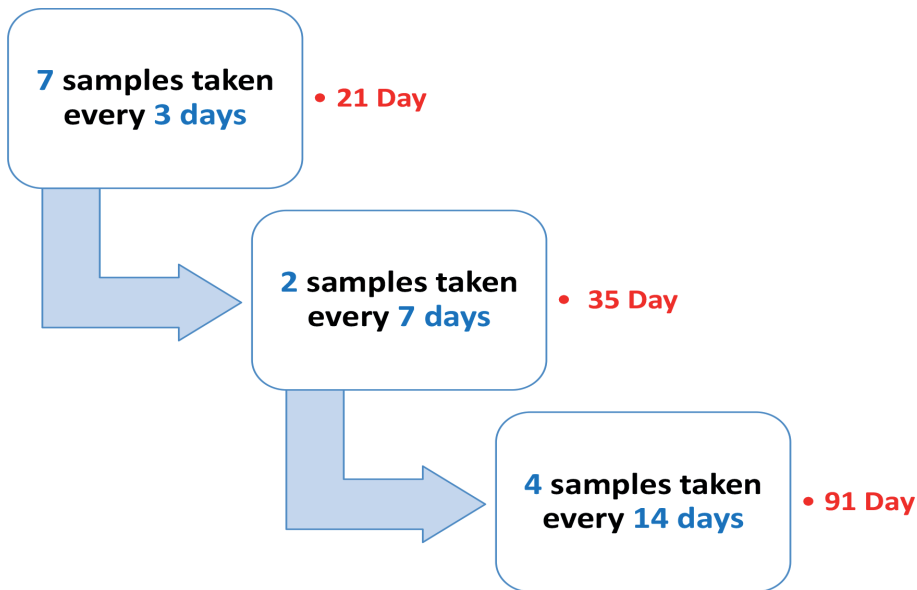


Figure 7.
Leaching test in a tank or tank with renewal of the lixiviant.

fills the volume more compactly [19]. Another work dedicated to the study of the behavior of pyrrhotite ash towards the environment has been published [21]. The main steps (**Figure 7**) and results (**Figure 8**) of this environmental study are shown below.

The test protocol is taken from the Dutch Standard NEN 7345 adapted to the Moroccan hydrological context. (MBMD: Modified Building Materials Decree) [1]. It is a Standard used to highlight the polluting potential of the fly ash recovered in construction materials.

Test protocol and conditions are the following:

- Spring ambient temperature;
- Bricks of dimensions 35 * 15 * 70 mm, aged for 28 days;
- Pure water as a washing agent with a ratio $V_L / V_S = 3.00$, (V_L : Volume of water, V_S : Volume of brick).

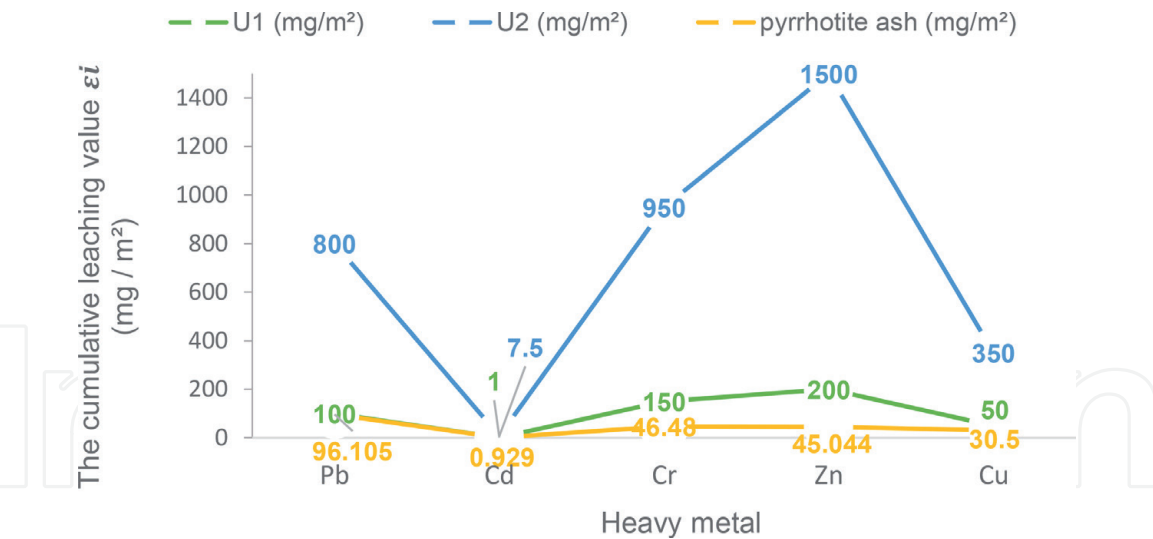


Figure 8.
The cumulative results of the heavy metals studied.

The leachates obtained were filtered using a 0.45 μm membrane filter, and after measuring the pH, they were acidified to pH 0.9–1.1 with a concentrated HNO_3 solution. After determining the concentration of heavy metals (Cr, Zn, Cd, Pb and Cu) using an Inductively Coupled Plasma Spectrometry, the ϵ_i ($\Sigma\text{mg} / \text{m}^2$) value was calculated and compared with limits U_1 and U_2 to identify the category of this waste. The **Figure 8** shows that all cumulative of the heavy metals studied are below the limit U_1 [26–28], so the pyrrhotite ash can be classified as waste that can be reused in construction materials.

4. Elaboration of yellow clay-based bricks

Currently, the unique application of the yellow clay is the pottery. So, this study aims to valorize the yellow clay differently. The feasibility to elaborate bricks for construction based on the yellow clay was more detailed in an article previously published [22].

4.1 The effect of adding a mineral waste: case of pyrrhotite ash

The study of bricks treated at 1000°C shows that substituting up to 30 wt.% natural clay with pyrrhotite ash has a positive effect on the technological properties of bricks.

A notable variation of all these properties was observed namely: shrinkage, weight loss, porosity, bulk density, water absorption capacity and mechanical strength. **Table 4** summarizes the results obtained for the brick containing 0 wt.%, 20 wt.%, 30 wt.% and 40 wt.% ash as well as the requirements of certain standards for building bricks available in the literature.

In order to better respond to the imperatives of the sustainable development that requires the respect of the environment and the rational use of resources and energy, it was deemed necessary to further study the manufacture of bricks-based clay and ash by seeking to optimize the manufacturing conditions using minimum of energy. For this reason, the evaluation of the technological properties of bricks containing ash was also made for bricks fired at 900°C. The **Table 5** summarizes results of this study.

At 900°C, the brick containing 20 wt.% of pyrrhotite ash exhibits the best flexural strength (28.69 MPa). Since it reveals a low porosity, it was decided to improve

Test	Standard	Requirement	Bricks with 0 wt.% of ash	Bricks with 20 wt.% of ash	Bricks with 30 wt.% of ash	Bricks with 40 wt.% of ash
Shrinkage (%)	ASTM et CNS 382.R2002	< 8	10	6	5	2
Weight loss (%)	CNS 382 (Chinese National Standard)	<15	20	11.98	11.13	10
Porosity (%)	SIA 266 ASTM C62	[20–55] <40	36	36.41	39.4	42.11
Water absorption (%)	ASTM C62	< 22	13.8	16.16	21.6	23.2
Bulk density (g/cm ³)	NF P 94–093	[1.5–1.8]	1.63	1.79	1.87	2.04
Flexural strength (Mpa)	EN 771–1	> 7	7.65	23.88	31.16	28.53

Table 4.
Summary of the clay bricks study with 0, 20, 30 and 40 wt.% content of pyrrhotite ash fired at 1000°C.

Percentage	Shrinkage %	Weight loss %	Porosity %	Flexural strength MPa
20 wt. %	11	14.74	19.69	28.69
30 wt. %	10	13.94	20.38	18.05
Standards requirements	<8	<15	[20–55] and <40	>7

Table 5.
Summary of the clay bricks study with 20 wt.% and 30 wt.% content of pyrrhotite ash fired at 900°C.

Temperature	Shrinkage %	Water absorption %	Porosity %	Density g/cm ³	Compressive strength MPa
900	1.34	20.65	50.14	1.806	11.82
1000	0.89	19.88	50.78	1.797	10.78
Standards requirements	<8	<22	[20–55]	[1.5–2]	>7

Table 6.
The technological properties of the best formulation of clay-ash-sawdust bricks fired at 900 and 1000°C.

this formula (20 wt.% of pyrrhotite ash – 80 wt.% of yellow clay), by adding an organic element which evaporates during firing, leaving pores which will lighten the brick thus produced. For this purpose, cedar sawdust was chosen to be added to yellow clay bricks incorporating 20 wt. % of pyrrhotite ash.

4.2 The effect of adding an organic waste: case of cedar sawdust

Table 6 shows that bricks containing 20 wt.% of pyrrhotite ash, 80 wt.% of clay and 5 wt.% of sawdust of the total mass has characteristics corresponding to

the requirements of international standards for terracotta bricks, whether fired at 900°C or 1000°C. However, those fired at 900°C are more resistant to compression with a value of 11.82 MPa against 10.78 MPa for those fired at 1000°C.

5. Discussion

The technological evaluation of bricks containing pyrrhotite ash was mainly carried out by measuring shrinkage, weight loss, bulk density, porosity, dilatometry and mechanical strength.

About bricks containing yellow clay and pyrrhotite ash, the results (**Table 4**) shows that the addition of the mineral waste increases the bulk density and flexural strength. However, the addition of up to 30 wt.% of waste enhances the flexural strength. This property decreases when 40 wt.% of waste is added. Bricks containing pyrrhotite ash up to 30 wt.% exhibits a good bulk density with an increase of the flexural strength. Such a behavior was attributed in our previous work [22] to:

- The low decomposition of organic matter from the waste sample [19], thus low generating pores in the fired structure;
- The presence of a high content of iron particles in the waste sample, which do not induce flaws in the fired ceramic matrix;
- The high density of waste particles [19].

The maximum of flexural strength is obtained for the brick containing 30 wt.% of waste. The flexural strength decreases for bricks containing more than 30 wt.% of waste. This behavior was attributed in our previous work [20] to the firing temperature that should be higher than 1000°C, as well as the complex mineralogical transformations, occurring during the firing and cooling process, that depend on the content of Fe_2O_3 and fluxing oxides.

The evolution of porosity is in accordance with the water absorption values but is not in accordance with the bulk density and flexural strength values especially for the percentage of 40 wt.%. This behavior was explained in our previous work [22] by the fact that the increase of the waste content causes the coalescence of pores which leads to generating regions of weakness, which, in turn, weakened the mechanical properties of the bricks when the content of pyrrhotite ash is higher than 30 wt.%.

The bricks with 20 wt.% of pyrrhotite ash and 80 wt.% of clay - fired at 900°C - have the best mechanical properties with a flexural strength of the order of 28.69 MPa (**Table 5**). Knowing that the bricks intended for construction must have a mechanical strength which exceeds 7 MPa [29]. However, this type of bricks have a shrinkage of 11% (which exceeds the limit <8%) [30], a weight loss near the limit 14.75% (<15%) [31] and also a porosity around the lower limit 19.6% (> 20%), which makes the bricks with 20 wt.% of pyrrhotite ash and 80 wt.% of clay dense and heavy. In order to improve all these properties, another element has been added to bricks containing 20 wt.% of pyrrhotite ash. This element should promote the formation of pores, which will reduce the bulk density of the bricks and obviously make them lighter. To remain around the concept of recycling waste to protect and serve the environment, the element chosen is an organic waste which is cedar sawdust from the artisanal sector in the city of Fez.

Adding sawdust to the bricks has the effect of reducing their weight so that the brick becomes light. The addition of organic waste results in light bricks or porous

bricks. The bricks become more porous [20% -55%] and meet European standards [15, 30] and the compressive strength remains above 7 MPa.

Experience has shown that bricks with a sawdust content of up to 5 wt.% of the mixture consisting of 20 wt.% of pyrrhotite ash and 80 wt.% of clay treated at 900° C, exhibits compressive strength of the order of 11.82 MPa, a porosity of 50.14% and a bulk density of 1.8 g/cm³, and thus meet European standards for building bricks.

6. Conclusions and outlook

The main objective of this study has been satisfactorily achieved. Yellow clay used in pottery field so far was valorized in a field in high demand around the world, that of the building bricks. By adding mineral and organic wastes, pyrrhotite ash and sawdust, the technological properties of yellow clay based bricks were improved.

This study has demonstrated the feasibility of using by-products (pyrrhotite ash and sawdust) as a partial clay substitute in fired clay products. Waste's content affected all technological fired brick properties significantly. Based on the results of the physical-mechanical properties evaluations of the end products and the environmental evaluation industrial waste is recommended as a raw material (pyrrhotite ash and sawdust) in the manufacture of fired clay products. Use of this waste could have practical implications as a means of recycling and for achieving costs savings in brick production, as fewer raw clay materials would be required. Most importantly, the added value of this study is the reuse of two wastes at a time in a brick with good technological performance that should be economically and environmentally beneficial.

On the other hand, this study made it possible to determine the optimal formulation and favorable conditions for the production of bricks based on yellow clay and wastes. With the view to make it exploitable, it is necessary to apply it on an industrial scale to verify the reproducibility of the results obtained in the laboratory. Even to build a prototype (typical house) in order to study the durability of the bricks in real conditions while following the parameters influenced by the properties of the bricks, namely the climate and the air quality inside the typical house.

Finally, this work opens the way to lead the reflection in order to expand the range of products where it will be possible to valorize the yellow clay and develop other materials used in the fields of buildings and civil engineering.

Acknowledgements

This work would not have been possible without collaboration with universities at international level. Note that the majority of the experimental part was carried out in Spain and the rest between Morocco and Lithuania.

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