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Fired clay bricks using agricultural biomass wastes: Study and characterization

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HIGHLIGHTS

- Wheat straw, sunflower seed cake and olive stone flour have been incorporated into clay matrix.
- The influence of the grinding and incorporation rate of the additives have been studied.
- Physical, mechanical and thermal properties of the fired brick have been affected.

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ABSTRACT

The main objective of this study is to investigate the effects of the incorporation of renewable pore-forming agents on the properties of fired bricks. Different additives have been studied (wheat straw, sunflower seed cake and olive stone flour) at different grinding and incorporation rate.

Physical properties such as linear shrinkage, loss on ignition, bulk porosity, water absorption and bulk density have been measured. Mechanical and thermal performances have also been characterized. The incorporation of 4 wt.% of sunflower seed cake, with the lowest grinding, leads to the best compromise between mechanical and thermal results compared to the reference brick.

1. Introduction

Based on a simple manufacturing process and the use of cheap and abundant raw materials (clay, sand and water), clay bricks are one of the most used building materials. Indeed, they present interesting mechanical and thermal properties [1]. However, with the recent development of more reliable materials, bricks come across technical limits because of their weight and limited thermal resistance.

In a context of sustainability and with the always more restrictive environmental regulations, environmentally friendly materials recycling wastes and saving energy have to be developed. It has been proven in the literature [2–4] that the incorporation of by-products or wastes is an innovative and efficient way to generate pores in fired bricks. The described pore-forming agents can be divided into two categories: those issued from renewable resources (for example rice straw [5], processed waste tea [6], sawdust [7]) and those from mineral resources (such as slags [8] or marble residues [9]). It was therein shown that a small amount of additives (generally below 10 wt.%), that burns during the firing process, led to an increase of the brick porosity and so a decrease of the thermal conductivity. However, it was also observed that this creation of pores generally caused a significant decrease of the mechanical performances [10]. Compromises between mechanical

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and thermal properties have thus to be made in order to produce a competitive building material. Numerous pore-forming agents have been tested in the literature, most of the time in order to lighten the final brick, the thermal aspect being left aside.

In this work, we focused on the incorporation of new additives such as wheat straw, sunflower seed cake and olive stone flour which were chosen for their low cost, availability and close location. The last two materials were never incorporated into clay bricks formulation; the sunflower seed cake being mostly used for animal feeding or to develop insulation fiberboards [11] or injection-moldable composite materials [12]. The olive stone flour was, on the other hand, used for producing thermoplastics [13]. Wheat straw has been once used as pore-forming agent in a former project of our laboratory [14] but the results obtained were not enough optimized.

After characterizing the different raw materials (vegetable matter and clay), the influence of the nature of the pore-forming agent, their grinding and incorporation rate were studied. Finally, the physical (linear shrinkage, loss on ignition, porosity, water absorption and bulk density), mechanical (bending strength) and thermal properties of the new developed bricks were evaluated.

2. Materials and methods

2.1. Characterization of the brick raw materials

2.1.1. Clay characterization

The clay mixture used in this study was provided by TERREAL (Castelnaudary, France).

The elementary compositions were measured through energy dispersive X-ray spectroscopy (EDX, 15 kV and 10 nA).

2.1.2. Agricultural wastes characterization

The three types of biomasses used in this study (wheat straw, sunflower seed cake and olive stone flour) were selected as they are locally produced, readily available and cheap. Furthermore, their distinct composition and grinding should induce different behaviors during the firing process.

The wheat straw and sunflower seed cake were provided by the agricultural cooperative ARTERRIS (Castelnaudary, France) and the olive stone flour by BARDON ETS (Le Muy, France).

The biomasses were first chemically characterized. The dry matter content was determined according to the French standard NF V 03-903. The mineral content was determined according to the French standard NF V 03-322. The fiber content (cellulose, hemicelluloses and lignin) was determined using the ADF-NDF method [15–17]. The protein content was determined using the Kjeldahl method according to the French standard NF V 18-100 with a multiplying factor of 6.25 for the nitrogen percentage. The lipid content was determined after a Soxhlet extraction using cyclohexane, during 6 h, according to the French standard NF V 03-908. All the characterizations were performed in triplicate.

The thermogravimetric analysis (TGA) of the vegetable matters was performed using a simultaneous thermal analyzer (NETZSCH STA 449 F3 Jupiter). The measure consists into reporting the weight loss of the sample of about 200–500 mg, placed into a cylindrical crucible, with an increase of temperature up to 1100 °C at a rate of 10 °C/min. The results were then derived using the PeakFit software to obtain the differential curve of the thermogravimetric analysis (DTG), in order to identify the decomposition occurring in the samples.

The sorption and desorption behaviors of all the agricultural wastes were observed using a dynamic vapor sorption (DVS) Advantage System from Surface Measurement Systems (Alperton, UK). The apparatus uses an ultra sensitive balance capable of measuring changes in sample mass as low as 0.1 μg. Samples were equilibrated at a constant temperature and different relative humidities (from 0% to 90%). The changes in relative humidity were induced using mixtures of dry and moisture-saturated nitrogen flowing over the sample. At the beginning of each measurement, samples were dried under dry nitrogen. From the complete moisture sorption and desorption profile an isotherm was plotted.

The water absorption, also called swelling *ratio*, of the vegetable matters was also determined by soaking the biomass into an excess of water overnight and measuring the relative increase of the solid volume when saturated in water.

2.2. Samples preparation

The clay was first ground in order to obtain a powder with particles of about 3 mm. Wheat straw and sunflower seed cake were crushed and sieved at different grindings: <0.5 mm and [0.5,1.0] mm. The olive stone flour was industrially ground at about 50 μm. Different amounts (4 and 8 wt.%) of additives were then mixed with clay in a rolling mill to enhance homogeneity.

The required quantity of water (up to 22.2 wt.% depending on the formula) was added to obtain the desired humidity and plasticity that are necessary to avoid defects onto the structure during the process.

The samples were then molded by extrusion process in the form of tablets ($175 \times 79 \times 17$ mm^3), dried up to 105 °C and finally fired up to 920 °C for 1 h, according to the industrial recommendations.

Samples were prepared and designated as Ref. for the bricks without waste and x-ByP-g for the mixtures with x the content of additives incorporated (in wt.%), ByP the by-product used (ByP = WS (wheat straw), ByP = SSC (sunflower seed cake) and ByP = OSF (olive stone flour)) and g the grinding of the biomass (in mm).

The sample composition is presented in Table 1.

2.3. Characterization of the bricks

The physical (linear shrinkage, loss on ignition, density, water absorption and porosity), mechanical (bending strengths) and thermal properties of the obtained clay bricks were determined.

Linear shrinkage was determined by measuring the length of the sample before and after drying using a caliper according to the standard ASTM C210-95. Loss on ignition was determined by measuring the mass loss of the sample between the drying and firing steps. Water absorption of these lightweight bricks was determined using the standard procedure ASTM C 373-88. The samples were dried at 110 °C for 24 h and weighed to constant mass. They were then cooled for 24 h and totally immersed in water. After soaking for 24 h, they were dried and reweighed to constant mass.

The bulk porosity and saturated density were determined according to the test procedure recommended by Hornain et al. [18] by means of water saturation under vacuum.

Thermal conductivity was obtained through a heat flux meter method. This method followed the standards ASTM C518, ISO 8301 and NF EN 12667. The measurement area was 60x40 mm^2 with a thickness of the sample greater than 10 mm. This apparatus produced a temperature gradient along the thickness of the sample and measured heat flux that gave through the software the thermal effusivity. Thermal conductivity was deduced using the formula: $\lambda = \frac{E^2}{\rho C_p}$ (W/m K) with ρ the bulk density (kg/m^3) and C_p the specific heat capacity (J/kg C).

All the characterizations were performed on 6 samples; the coefficient of variation of all the obtained values, defined as the *ratio* of the standard deviation to the mean, is verified to be prior to 5%, showing the accuracy of the presented data.

3. Measurements and results

3.1. Clay characterization

In Table 2 is reported the chemical composition of the fired clay. From these data, it is apparent that this clay is mainly composed of quartz (SiO_2), aluminum oxide (Al_2O_3) and hematite (Fe_2O_3). Calcium, potassium and magnesium oxides are present but to a less extent. Traces of titanium, sodium and phosphorus oxides were also observed.

Table 1
Sample compositions.

Sample	Ref.	4-WS-<0.5	8-WS-<0.5	4-WS-0.5/1	4-SSC-<0.5	4-SSC-0.5/1	4-OSF	8-OSF
WS (wt.%)	–	4	8	4	–	–	–	–
SSC (wt.%)	–	–	–	–	4	4	–	–
OSF (wt.%)	–	–	–	–	–	–	4	8
Water (wt.%)	15.9	19.1	22.2	19.5	20.3	19.8	17.3	19.0