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Properties of fired clay bricks with incorporated biomasses: Cases of Olive Stone Flour and Wheat Straw residues

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HIGHLIGHT S

• Modification of bulk density, porosity and thermal conductivity of fired clay bricks with organic matter.

• Technological impact of pore forming agents on shrinkage, loss of ignition.

• Mechanical loss due to porosity created by fired organic matter.

ARTICLE INF O

Keywords: **Fired clay bricks Organic matter Thermal conductivity Compressive and bending strength Porosity and bulk density**

ABSTRACT

In recent years, interest in green building materials and the valorisation of by products from multipl^e industries has been increasing. As the brick industry allows various compounds to be added during the mix ing procedure, much research has been conducted to highlight the impact of additions on fired clay bricks. This paper examines the significance of adding organic matter coming from agricultural solid waste (Oliv^e Stone Flour, OSF, and Wheat Straw, WS residues) to improve thermal performance white maintaining load be a ring capacity. The resul ts show a decreasing bulk den si ty for mixtures containingOSF, ranging from 6% to 19% compared to clay alone, and for WS mixtures, where the bulk density reduction is from 4% to

20%. Total porosityincreases by 5 56% forOSF, and by 7 67% forWS, implying lower thermal conductivity forWS (23% relative to clay atone) compared toOSF(16%)when 5%.wt is incorporated. The compressiv^e strength, for 5%.wtWS and 5%.wt OSF is 52% and 31% respectively. There is a significant positive corr^e lation between the increasing amount of organic matter and the porosity. The most striking result to ^ernerg^e from the data is that, for the sarne %.wt, WS creates higher total pore volume than OSF owing to the differ ence of grain size distribution. Consequently the pore size distribution of new materials containing OSF is **more structured and leads to a better compressive strength than WS.**

1. Introduction

Building sector produces almost 50% of worldwide carbon diox ide emissions. ln order to minimize this impact on the natural environment and human health, the use of water, energy and nat ural resources has changed by taking into consideration the entire life cycle of a product needs.

The building industry has called upon scientists to develop more sustainable, low cost and lightweight construction materials.

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Recently, some researchers $\begin{bmatrix} 1 & 6 \end{bmatrix}$ have reviewed the use of various wastes in brick production to achieve lightweight materials that are environmentally friendly white respecting the requirements of the relevant standards. A large variety of additives have been incorporated into bricks at laboratory scale.

The industrial process for brick production involves an extru sion stage where clay is mixed with different additives before being dried and fired. During the combustion, the biomass provides supplementary energy through its organic substances. Mostly, the incorporated substances do not exœed 10%.wt with an acceptable gain in porosity and thermal conductivity and mechanical resis tance is thus maintained [7 9].

The most common result obtained by the scientific community in this field is a decrease in bulk density. This physical property is

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enhanced by higher firing temperatures (over 1000 \degree C, [10]), water absorption increases positively with the amount of incorporated matter, depending on the nature of the additive, the particle size and the porosity generated [5].

Since brick industries have to declare the thermal conductivity of their products according to the standards in force (ISO 8301, IS08302 and EN 1745), this property has given rise to competition among the manufacturers of construction materials to achieve the best thermal insulation. The thermal conductivity of fired clay bod ies is commonly correlated with their density and total porosity. However, mineralogical composition and microstructure are con sidered to be important factors governing the thermal behavior of fired clay bricks [11 15].

In general, a decrease in density causes a fall in the thermal and mechanical values of clay bodies. During the firing phase, increasing temperature induces a lower bulk density and a higher thermal con ductivity due to microstructural modifications [14]. Regression analyses by Dondi et al. cast light upon a correlation between effec tive thermal conductivity and open porosity, quartz and Ca rich sil icates composing the clay bodies [11]: unlike the presence of quartz and Ca silicates, open porosity has a positive effect on insulation.

In most cases, compressive strength decreases with the amount of additives increases. As the mechanical behavior is related to porosity, few types of incorporation lead to improved load bearing. Waste tea, coffee grounds and boron waste have shown interesting results for firing temperatures over 900 \degree C [5,16 18].

The aim of this paper is to evaluate the physical and mechanical properties of construction materials manufactured by adding Olive Stone Flour (OSF) and Wheat Straw (WS) residues to the clay body. These two organic matters were chosen for their availability near the brick manufacture and never been incorporated into fired clay bricks at an industrial scale.

This work seeks to address the following questions: How do the nature, particle size distribution and the amount (%.wt) of OSF and WS incorporated modify the performance of fired clay bricks and how can industry move from the laboratory scale to the scale required for commercialized products?

Numerous studies have been conducted but few have been applied at industrial scale because of a lack of relevant standards, the difficulty of conserving waste materials, and doubts as to the willingness of the public to accept the use of waste materials in alternative bricks.

2. Materials and methods

The present experimental campaign was performed using OSF (2, 4, 5 and 8%. wt) and WS (1, 3, 5 and 7%.wt). The most appropriate method for this investigation was to explore physical properties such as density, porosity and thermal conductivity on extruded cuboid blocks 170 mm \times 75 mm \times 17 mm. The mechanical performance was obtained by uniaxial compressive loading on extruded hollow bricks 75 mm \times 80 mm \times 75 mm (thickness of internal layers 12 mm) to approach the actual commercial products.

2.1. Raw materials

The clay used was provided by TERREAL, a national ceramic industry. The clay paste was a mixture of 69% clay (illite and kaolinite) and 31% sand temper with 16%. wt water. The two additives were chosen for their different natures: Wheat Straw is crushed and sieved to obtain particles size inferior to 500 lm and Olive Stone Flour is delivered by the industrial with particles size of 50 μ m. These biomasses have been characterized in a previous work [19].

2.2. Preparation of samples

The ceramic body was prepared for extrusion by mixing the clay ingredients (clays and sand) with WS and OSF, water being added for plasticity (for good fabrication conditions) depending on the amount and type of organic matter incorporated.

This amount of water was adapted to the extrusion pressure and to achieve homogeneity for the two types of sample (alveolar bricks and solid blocks). Extruded samples were dried at temperatures up to 105 °C and fired in an electric

furnace at up to 920 °C for 1 h. The preparation of laboratory scale products was conducted by the industrial in order to keep the same protocol used for commercialized products.

2.3. Samples characterization

Bricks were characterized at our laboratory according to technical standards for water absorption, total shrinkage, loss of ignition and bending strength, while porosity, density, thermal conductivity and compressive strength tests were performed on fired bodies.

All the tests were carried out on 6 samples for each formula.

Water absorption (WA) for lightweight bricks was determined in accordance with ASTM C 373-88. Six bricks per composition were dried to constant mass and cooled before being placed in a water tank at room temperature $(20 °C)$. After 24 h, the specimens were removed from the tank and the surface water was wiped off. The water absorption of each specimen was calculated using Eq. (1):

WA
$$
(\%)
$$
 $\frac{m_w - m_d}{m_d} \times 100$ $\begin{vmatrix} m_d \text{ mass of oven dried specimens (g)} \\ m_w \text{ mass of wet specimens (g)} \end{vmatrix}$ (1)

Total shrinkage values were obtained by measuring the length of the samples with a calliper before and after the firing stage in accordance to standard ASTM C210-95 and using Eq. (2):

ST
$$
(\%)
$$
 $\frac{l_p}{l_p} \times 100 \begin{vmatrix} l_p \text{ specimen plastic length (mm)} \\ l_f \text{ specimen fired length (mm)} \end{vmatrix}$ (2)

Loss of ignition (LOI) was determined by measuring the mass loss of the sample between the drying and firing stage following Eq. (3):

LOI (
$$
\%
$$
) $\frac{m_d - m_f}{m_d} \times 100 \bigg|_{m_f}$ mass of oven dried specimens (g) (3)

Plasticity, P_s , is an important parameter characterizing the clay deformation. This parameter is controlled to avoid extrusion failures and heterogeneities.

$$
P_S (\%) \quad \frac{m_w - m_d}{m_d} \times 100 \Big| \frac{m_w \text{ wet state weight of the sample (g)}}{m_d \text{ mass of oven dried specimens (g)}} \tag{4}
$$

Bulk density and water accessible porosity were determined by the gravity method [20], using vacuum saturation. Once sample are fully saturated, the volume is determined by hydrostatic weighing. Then, samples are submitted to oven drying at a temperature of 105 ± 5 °C until the weight of the sample remains constant.

Porosity, ε , is determined using the following equation:

$$
\varepsilon(\%) \quad \frac{m_{\text{air}} - m_{\text{dry}}}{m_{\text{air}} - m_{\text{water}}}\Big|_{m_{\text{air}}}^{m_{\text{dry}}} \text{master saturated mass sample (kg)}
$$
\n
$$
\varepsilon(\%)
$$
\n
$$
\varepsilon(\%)
$$
\n
$$
\frac{m_{\text{air}} - m_{\text{water}}}{m_{\text{water}}} \text{Archimedes mass sample (kg)}
$$

 (5)

Bulk density, ρ , is determined using the following equation:

 $\left|m_{\text{dry}}\right|$ mass of fired sample dried at 105 °C until stabilization $\left(\text{kg}\right)$

$$
\rho(\text{kg/m}^3) \quad \frac{m_{\text{dry}}}{m_{\text{air}} - m_{\text{water}}} \times \rho_w \begin{vmatrix} m_{\text{air}} \text{ water-saturated mass sample (kg)} \\ m_{\text{water}} \text{ Archimedes mass sample (kg)} \\ \rho_w \text{ water density (kg/m}^3) \end{vmatrix}
$$
 (6)

Open porosity and porosimetric distribution were determined by mercury intrusion porosimetry (Thermo Finnigan Pascal 140/240) with an experimental uncertainty of about 1% relative. The two-stage MIP experiments were performed on samples of 1 cm³ were extracted on cuboid fired clay blocks and oven dried during 24 h at 105 \pm 5 °C. The pore diameter is calculated according to Washburn law described in the following equation:

$$
D(m) \quad \begin{array}{c} \begin{array}{c} \text{D pore diameter (m)} \\ \text{P pressure of intrusion of the mercury (Pa)} \end{array} \\ \begin{array}{c} \begin{array}{c} \text{A} \gamma \cos \theta \\ \gamma \sin \theta \cos \theta \end{array} \\ \begin{array}{c} \end{array} \end{array} \begin{array}{c} \text{P pressure of intrusion of the mercury (Pa)} \\ \text{N} \end{array} \\ \begin{array}{c} \end{array} \end{array} \begin{array}{c} \text{A} \end{array} \end{array}
$$

Thermal conductivity was obtained by an indirect method using thermal effusivity E (asymmetric hot plate method: DESPROTHERM; Eq. (8)) and tabulated values using dry density according to table in annex A of European standard EN 1745.

Samples were cuted on cuboid fired clay bricks to fit to the required equipment size of 60 mm \times 40 mm. The parallel faces were polished and grinded to avoid default contact with the platens. The thickness $(\sim 10 \text{ mm})$ should ensure that heat flow did not pass through the sample during the measurement. Samples were oven dried during 24 h at 105 ± 5 °C then cooled in a controlled ambiance 20 °C, 50% relative humidity.

$$
\lambda(W/m.K) = \frac{E^2}{\rho \times c_p} \begin{vmatrix} E \text{ thermal effusivity } (J/m^2 \text{ K s}^{1/2}) \\ \rho \text{ dry density } (kg/m^3) \\ C_p \text{ estimated heat capacity value } (J \text{ kg}^{-1} \text{ K}^{-1}) \end{vmatrix}
$$
(8)

The bending strength of the fired samples was determined by three-point bending in a mechanical testing machine with a loading rate of 0.5 kN/s until failure at F_{max} (N). Loading was applied to cuboid blocks at a constant rate. Bending stress was obtained through the following equation:

$$
\sigma_f(MPa) = \frac{3 \times F_{\text{max}} \times l}{2 \times b \times h^2} \begin{vmatrix} b & \text{depth} & 75 \text{ mm} \\ h & \text{width} & 17 \text{ mm} \\ l & \text{support span} & 150 \text{ mm} \\ F_{\text{max}} & \text{failure force (N)} \end{vmatrix} \tag{9}
$$

The compressive test was carried out on extruded hollow bricks 75 mm \times 80 mm \times 75 mm (thickness of internal layers 12 mm) using a 4000 KN hydraulic press with a loading rate of 0.5 kN/s. The bricks were loaded in the direction of the hollows, over the cross sectional area S (mm²). The compressive strength was calculated using the Eq. (10):

$$
\sigma_c(MPa) \quad \frac{F_{\text{max}}}{S} \bigg|_{F_{\text{max}}}^{S} \text{ surface area } (\text{mm}^2) \tag{10}
$$

The surfaces in contact with the platens were surfaced on a grinding machine in order to obtain correct parallelism according to the specifications of the standards [21,22].

For the calculation, the overall dimensions were measured. The effective surface was measured by including alveolar areas.

3. Results and discussion

3.1. Physical, hydric and thermal properties

3.1.1. Water absorption (WA)

Water absorption is a durability indicator; it gives information about open porosity. Low values imply good resistance to the nat ural environment and acceptable permeability of bricks. From the results in Table 1, it is clear that increasing the rate of organic incorporation led to higher WA than in the clay reference, which can be explained by the difference in the open porosity of the 5 mixtures. For comparable %.wt incorporation and %.wt of sand, WS5 water absorption was 18% higher than that of OSF5.

3.1.2. Firing linear shrinkage (ST)

Shrinkage was directly affected by firing temperature. All the for mulations studied in this paper were prepared following the same process (extrusion, drying and firing at $920 °C$). Shrinkage occurs when the chemically and mechanically bound water evaporates, so the temperature and time of the drying and firing stages must be controlled [23]. Shrinkage during the ceramic process is a signif icant parameter, since structural change and solidification, implying densification, may create tensions and failures in fired clay bricks [24]. During this process, open porosity is transformed into closed porosity. According to the literature, this value must be below 8% and the results presented in Table 1 are in accordance with requirements.

WS formulations present lower firing shrinkage (measured at 920 \degree C) than the clay reference and OSF formulations. The ST val

Table 1

Water absorption (WA), firing linear shrinkage (ST), loss of ignition (LOI) and plasticity (Ps).

Designation	WA (%)	ST(%)	LOI(%)	Ps(%)
Clay	13.6	5.9	9.2	16.2
W _{S1}	15.3	5.2	10.4	17.0
WS3	18.9	4.8	11.9	18.6
WS ₅	24.7	4.2	13.2	20.2
WS7	28.6	3.8	14.9	21.3
OSF ₂	15.2	5.8	11.3	17.0
OSF4	17.9	5.9	13.0	17.6
OSE ₅	20.2	5.7	13.8	17.6
OSF ₈	26.3	5.5	17.6	18.8

ues obtained for WS are in descending order as the amount of WS rises. Addition of RH in fired clay bricks decreased the drying and firing shrinkage for the bricks. The OSF shrinkage value was close to that of the clay reference (5.9%) even for the high amounts OSF5 and OSF8 (resp. 5.7% and 5.5%).

3.1.3. Loss of ignition (LOI)

The loss of ignition represents the brick mass diminution as a consequence of dehydroxylation reactions, carbonates decomposi tion, water evaporation and combustion of biomass residues. This loss is related with the creation of pores in the fired brick.

The clay reference has an LOI of 9.2%. A comparison of the WS and OSF formulations reported in Table 1 indicates that the OSF LOI is likely to increase as the quantity of incorporated biomass becomes greater. The same trend is observed for WS. The higher the loss of ignition, the higher the water absorption. This is to be expected as described below. The porosity created should be corre lated with LOI and increase in the same way.

3.1.4. Plasticity (P_s)

Plasticity of the clay/biomass mixture increases with the amount of additives. It is also observed that the results depend on the nature of the biomass. For Wheat Straw that contains more cellulose fiber the water needed is more important as the WS absorbs water through its natural existing water channel [25].

3.1.5. Density and porosity

Apparent density, water accessible porosity and mercury intru sion experiments analysis on the 6 formulations are given in Table 2.

Obviously, the addition of organic matter increases the brick porosity and decreases its density [2,3,5,6,18].

In comparison with the clay reference, WS5 shows a drop of 16% for density and a gain of 52% concerning porosity accessible to water. OSF5 is 12.5% less dense than clay and its porosity is 33% higher.

Fig. 1 describes the pore size distributions for the 8 formula tions compared to the clay mix.

Increasing %.wt of Wheat Straw generates greater pore volume compared to Olive Stone Flour. In comparison with clay pore size distribution, OSF formulas are very similar to clay reference. The smallest pores are associated with fine clay particles (Illite and kaolinite) and organic matter decarbonatation. As the fraction of sand (5%.wt and 10%.wt) is adapted to the amount of WS and OSF, quartz fraction volume contributes to larger pores.

Water absorption for OSF indicates that the porosity may be more closed and connected than that of WS. This can explain why the bulk density diminishes with OSF but the water absorp tion is not affected as much.

Fig. 1. Pore size distributions for Wheat Straw and Olive Stone Flour compositions.

3.1.6. Thermal conductivity

The thermal conductivity of the materials used plays a key role in avoiding heat loss and conserving energy. As the bulk density and porosity are the major factors governing thermal conductivity

[14,26 29], a great number of studies has focused on lightweight fired clay products obtained by adding pore forming agents.

For the clay body reference, the thermal conductivity was found to be 0.51 W/m K with an experimental method and 0.55 W/m K

Fig. 2. Thermal conductivity vs. dry density.

Table 4 Mechanical properties of fired clay bricks.

Designation	Fired-bending strength (MPa)	Compressive strength (MPa)
Clay	12.95	44.4 ± 1.3
WS1	13.37	$36.2 + 1.6$
WS3	12.42	$26.6 + 1.4$
WS5	8.40	$21.4 + 1.0$
WS7	7.10	$18.1 + 0.2$
OSF ₂	14.22	34.0 ± 1.2
OSF4	12.81	31.5 ± 2.7
OSF ₅	11.02	30.5 ± 1.2
OSF ₈	8.13	$748 + 36$

according to the standard [30]. As shown in Table 3, the higher the porosity the lower thermal conductivity $[11]$. The sizes of the pores and total pore volume created by WS and OSF are quite different and thus materials of very similar densities (WS7 and OSF8 for example) can have different thermal conductivities, with OSF lead ing to higher values than WS.

The percentage of sand is another factor that may affect thermal results, given that quartz has high thermal conductivity [31]. No light could be shed on the question in the present study because of a lack of formulations with different amounts of sand for the same quantity of incorporated organic matter.

The values were measured across the direction of extrusion, which could affect the observed thermal conductivity as the clay and organic matter tended to orient themselves parallel to the extrusion axis. This may show anisotropic behavior of ceramic

bodies, where the thermal conductivity parallel to the extrusion direction is higher [32]. This anisotropic aspect has also been con firmed on unfired clay bricks: thermal and hygrothermal proper ties were depending on tested direction and showed the impact of the clay layers orientation [33].

Fig. 2 describes thermal conductivity trends according to the tabulated values of annex A in the standard [30] and to experimen tal measurements obtained with a hot plate apparatus.

The values given in the standard are slightly lower (maximum difference of 15%) compared to experimental data, except for WS5.

Considering the data from the standard, the lowest values were obtained for WS7 (\sim 0.30 W/m K) and OSF8 (\sim 0.39 W/m K), with a gain of +30% compared to the clay reference, while the hot plate apparatus gave the lowest thermal conductivity for WS7.

Overall, the incorporation of organics reduced the thermal con ductivity of fired clay samples.

The differences in porous texture of WS and OSF influenced the thermal results, with OSF barely affecting thermal conductivity compared to WS incorporations. This indicates the influence of total porosity on thermal conductivity in this case.

3.2. Mechanical properties of dried and fired formulations

To investigate the load bearing capacity of the building materials including the WS and OSF additions, a three point bending test was performed on extruded bricks of dimensions $170 \text{ mm} \times$ 75 mm \times 17 mm while a uniaxial compressive loading test was

Fig. 3. Compressive strength vs. porosity.

Fig. 4. Relationship between CS and %.wt of organic matter.

Table 5 Coefficient *a,* **coefficient of correlation and standard error for 95% confidence level.**

	Standard Error	Coefficient of determination (r^2)	Akaike's information criteria (AICC)	a		
				Value	Std error	Range of confidence (95%)
WS	0.47	0.999	-1.75	4.76	0.187	$2.38 - 7.13$
OSF	0.17	0.999	-8.01	10.25	0.162	$8.18 - 12.32$

carried out on extruded hollow bricks 75 mm \times 80 mm \times 75 mm. The results are reported in Table 4.

Over 3% by weight of biomasses incorporation both density and mechanical strength decrease, while water absorption increases. Weakening of the mechanical strength is mostly due to increased porosity and microstructural tlaw size, particularly when large amounts of big particles are incorporated (WS7 for example). How ever, WS1, and particularly OSF2, bending strength values increase slightly compared to the clay reference. This improvement may occur when grains and pores are distributed homogeneously in the clay matrix. According to the literature, the increased mechan ical strength stems from new crystalline phases created (especially unreacted MgO) during the firing step [31).

Decreasing density, allowing good lower thermal conductivity, inevitably cornes at the cost of reduced mechanical strength.

The results obtained for OSF were slightly higher than those for WS. It is clear that compressive strength is related not only to the density of samples but also to their porosity and pore size distribu tions $[25,35,36]$.

Linear regression with the least squares method was used to determine the function giving the normalized mean compressive strength for each sample according to the amount of incorporated organic matter.

Predictive analyses describing the evolution of compression strength as a function of %.wt of incorporated matter are plotted in Fig. 4. We have used an harmonic decline (type: regression), statistical analysis is displayed in Table 5 and show good agree ment between predictive equation ($cf.$ Eq. (11)) and experimental data within the range of 0 9% of organic matter.

 $\sigma_{c,n}$ compressive strength for formulation containing $x\%$ of organic matter (5 and 10%.wt of sand)

 σ_{c0} initial compressive strength of clay without additives (30% wt of sand) (11) \times percentage of organic matter

a coefficient depending on the nature of the organic matter added

A number of authors have reported a decrease of compressive strength with increasing additive percentage. Sorne improvements in firing time and temperature may yield compact ceramic bodies with acceptable compressive strength [34).

The most critical values were obtained for WS7 and OSF8 with a decrease of 59% and 44% respectively. From the chart in Fig. 3 link ing total porosity and compressive strength, two different slopes can be observed for WS and OSF.

4. Conclusions

This paper proposes sustainable solutions for high thermal insulation by using fired clay bricks obtained by substitution of clay materials with different types of biomass (WS and OSF). This can provide a solution for recycling agricultural waste while con serving load bearing capacity.

From the technological tests, it was observed that both WS and OSF used at a percentage of 5%.wt showed better results than WS7 and OSF8, while maintaining the compressive strength and accept able water absorption of brick samples. Their role as pore forming agents was confirmed through a weight reduction of 12 16%. The addition of biomass was still limited in order to reach equilibrium between weight decrease and porosity increase correlated to a water absorption and mechanical decrease until acceptable values compared to standards. This study needs to be completed with scanning electron microscope investigation to better understand how organic matter modifies ceramic properties, especially con cerning thermal conductivity.

Finally, the development of numerical models to predict the compressive strength of clay bricks should be of interest to manu facturers seeking to produce materials that comply with the spec ifications. The results presented should be regarded as specific only to the organic materials tested.

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