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Research paper

Development of sustainable fired clay bricks by adding kindling from vine shoot: Study of thermal and mechanical properties

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This paper studies the addition of kindling from vine shoots in the production of fired clay bricks in order to achieve a better insulation of the buildings enclosure and a new way for recycling vine shoots, a waste which is widely produced in vineyards. Therefore, the influence of kindling addition on the thermal and mechanical properties of the fired clay bricks has been investigated in partnership with a local brick factory.
As result, it could be concluded that the amount of kindling that can be added is around 11%, whereby the brick's mechanical and physical properties abide by settled regulations for structural clay bricks, in accordance with current regulations. The added waste has improved bricks conductivity properties by reducing it up to 62% compared to the brick made without any waste. This means an improvement up to 34% for the equivalent thermal transmittance of a typical single-leaf wall assembly.

1. Introduction

Thermal energy consumption in buildings represents an important percentage of the total amount. In accordance with the European commission (2012), the residential and tertiary sectors account for approximately 40% of the final energy consumption in EU-28 and 36% of the greenhouse gas emissions. Despite the growing trend of number of buildings constructed on the last decade shows a high slowdown, the European Commission considers building sector as the one with the greatest energy-saving potential. This includes both, new buildings and major renovations of the old ones. Furthermore, the total amount of energy spent in a building is mainly determined by enclosure's quality and target thermal comfort parameters (Lecuona Neumann et al., 2005).

The characteristics of the material forming the building enclosure are vitally important since they constitute the barrier that separates and protects them from outside, determining the thermal indoor comfort. Their properties shall determine the building's energy demand and, therefore, the ensuing environmental impact (Theodosiou and Papadopoulos, 2008).

The present work deals with improvement of the building thermal insulation by reducing the amount of energy required for heating and air conditioning. There are several points of view on how to solve this problem and thereby many different ways to proceed. In this case, the project focuses on reducing the thermal conductivity (TC) of fired clay by adding agricultural wastes. This means obtaining the lowest equivalent thermal transmittance of the wall by developing new fired clay bricks made of new raw materials. Needless to say, it is mandatory to preserve the physical and mechanical properties required for different types of bricks, mostly if they are used with structural purposes.

The chosen waste, in this case, was kindling of vine shoots. Every winter the vines must be pruned. This gives rise to enormous quantities of a woody waste product that must be disposed of (Alfonso et al., 2009). Many ways of potentially making use of pruned vine shoots are currently at the technical viability testing stage (Ntalos and Grigoriou, 2002; Corcho-Corral et al., 2006; Luque et al., 2006; Ioannidou and Zabaniotou, 2007; Jiménez et al., 2008, 2009; Valente et al., 2010).

However, the use of this waste as bio-fuel requires a dry process that reduces the efficiency (Gañán et al., 2006; Mediavilla et al., 2009; Ruiz et al., 2013). This paper tries to contribute showing a new use in the ceramic sector, where moisture is automatically removed by process and does not involve an increasing of energy demand. Moreover, the vine shoot higher heating value contributes to the energy balance in the kiln and produces important saves on the firing process.

Several researches have been carried out lately, searching for new organic wastes from different kinds of wastes, in order to achieve ecological bricks (Devant et al., 2011; Muñoz Velasco et al., 2014). Although there are many papers relating experiences, they rarely focus on the thermal conductivity (Eliche-Quesada et al., 2011; Gökhan and Osman, 2013; De la Casa and Castro, 2014). Furthermore, some researches do not show the test method (Montero et al., 2009), do not

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follow any standard (Aeslina et al., 2010) or use equations to calculate it (Mucahit and Sedat, 2009).

As it must be, research has not to consider only scientific goals but also civil society goals. For this reason, this study took into account the economic and technical feasibility for investors and constructors, keeping in mind the environmental impact as well. Accordingly, the project was developed in partnership with Herederos Cerámica Sampedro S.A., a brick manufacturer located in Lardero, La Rioja-Spain and with the Servicio de Investigación y Desarrollo Tecnológico Agroalimentario del Gobierno de La Rioja ["La Rioja Regional Government Food and Agriculture Technological Research and Development Service"]. The aim is to contribute as a first approach to show a new way of use, for this waste, which is large produced, as a real chance for fired clay bricks manufacturing.

2. Materials

The specimens were formed trying to reproduce the usual manufacturing conditions, with special attention to the forming pressure and the firing temperatures, with a maximum of 950 °C. A scheme of the entire process planned is shown in Fig. 1. It has been highlighted both the processes developed in the partner factory and those that were carried out in university laboratories. Due to the high financial cost, the required energy and unnecessary environmental impact that involves the minimum clay load, settled by the industrial extruder (approx. 300 ton), clay was provided by our partner from the starting point of the process and then mixing with kindling and formed in the University facilities, and then specimens were inserted just before the drying process. From this point, the samples underwent the entire process until the end.

The vine shoot samples were obtained from the experimental vineyards run by the La Rioja Regional Government Food and Agriculture Technological Research and Development Service.

Once the raw material has been characterized, fractions of clay were mixed with different percentages of waste. Water was added until the consistency of the mixture allowed the forming of the specimens under the same pressure that is usually reached in the industrial process (250 kPa). Specimens were pressed in laboratory since the minimum load of the manufacturer's extruder is much higher than the amount required. Moreover, it has been reported that the forming method does not substantially influence the final brick performance (Gualtieri, 2010) since the pressure is the same.

Green (before firing) specimens were identified by a code scratched on the surface according to the amount of waste (Table 1). The selected percentage has been chosen based on the previous references but with both aims to complete the series and have a closed reference to compare results. Then green specimens were transported to the industrial facilities to enter the drying process and then went through the tunnel kiln. The drying process is based on several stages where temperature and humidity are under control and monitoring. In the first stage, air is settled at 25 °C and 90% humidity and, after 10 chambers, it ends at 100 °C and less than 5% humidity.

Once the drying is finished, an automatic process places the bricks on the wagons, and the firing stage starts. The setting points and control for firing process was based on the knowledge of the dilatometric curve of specimens.

3. Methods

3.1. Raw materials

First, kindling was characterized by determining the mean moisture. This was determined by heating the samples at 110 °C until constant weight is obtained. These data are necessary in order to mix the clay with the correct percentage of waste. The dry-matter was chipped and trammeled. Kindling particle size distribution was not measured, but maximum particle size was controlled and limited by using a sieve 1.40. Higher heating value (HHV) has been determined by using a semi-automatic isoperibol calorimeter made by LECO. Vine shoots were collected randomly and crushed, then 15 pellets were made by pressing, and finally they were dried into a muffle until constant weight was achieved. The HHV must be controlled in order to prevent an excessive local temperature peak in the samples that may produce cracks during firing due to the high difference in temperatures between zones of the samples.

The subsequent elemental analyses were carried out in the Regional Laboratory of the Government of La Rioja, accredited according to UNE-EN ISO/IEC 17025 and in the laboratories of the Departments of

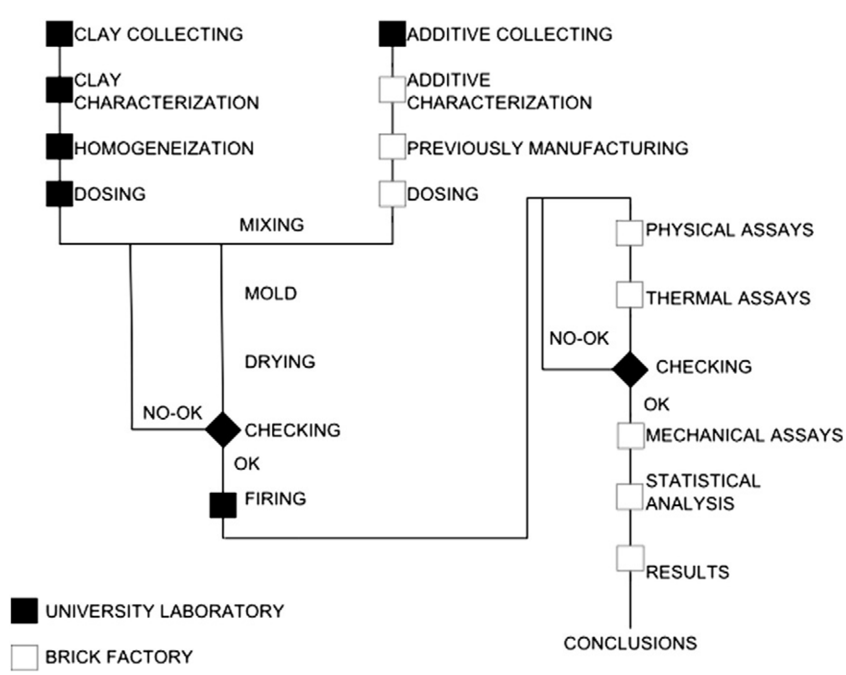

Fig. 1. Research plan scheme.

Chemistry and Mechanical Engineering of the University of La Rioja for kindling wastes.

In the case of clay, the dilatometric curves, obtained in accordance with UNE-EN ISO 10545-8 (2014) and chemical composition, were provided by Laboratorio Cerámico Sebastian Carpi, which is accredited by ENAC in accordance with UNE-EN ISO/IEC 17025 for the performance of test of ceramic tiles.

3.2. Thermal test

TC was measured by the determination of thermal resistance by means of guarded hot plate and heat flow meter method with a GUNT WL376 equipment. After a transitory state, the mean value of last 15 min (and once values were fully stabilized) was recorded and related to setting temperatures. These conductivity values are obtained for a temperature interval among the hot plate and the cold plate. The assay was repeated three times for each specimen, with three different temperature ranges, thereby giving three pairs of conductivity-mean temperature.

The determination of regression lines for each specimen enabled us to calculate conductivity value at 10 °C (λ_{10}, dry), according to EN 1745 (2002). In order to test the statistical significance of these values, the corresponding analysis of variance (ANOVA) was performed.

3.3. Mechanical tests

The testing machine (Servosis MES150) provided data about time, displacement and applied load in each assay. Since the dimensions of each specimen were known, the stress-strain curve was plotted and the CS determined according to EN 772-1 (2011).

As Rankine fracture criterion for brittle material states, the fracture is produced when one of the main strength tensions is above the compression strength. Therefore, it is expected that the ceramic block will withstand a CS equal to that obtained in the specimens assayed.

3.4. Physical tests

The addition of combustible solid particles to the clay leads to an increase in porosity (Cultrone et al., 2004). This higher porosity reduces the mass of the piece, which entails a decrease in density (Turgut and Yesilata, 2008). The specimens were weighed at dry state (W_D) as well as in water after 24 h (W_{SW}) and weighed again at the saturated wet state in air (W_{SA}), in order to determine the bulk density (BD) and the water absorption (WA) by calculation as it is indicated by EN 772-13 (2000), EN 772-7 (1998). In each step of the production line (forming-drying-firing), the specimens were weighed and measured in accordance with EN 772-16 (2011), and thus this linear shrinkage (LS) has been determined. These values are essential for an accurate design of extrusion dies.

Open porosity (AP) has been obtained by following UNE-EN 772-4 (1999). This standard leads to determine the total volume of pores which are in contact with the atmosphere directly or through others pores by the Eq. (1):

$$AP[\%] = \frac{W_{SA}-W_D}{W_{SA}-W_{SW}} \cdot 100 \tag{1}$$

Table 1
Composition series.

Batch [code]	Waste [wt%]	Mixing water [wt%]
AA00	–	20%
AS05	5%	20%
AS11	11%	20%
AS17	17%	20%

Table 2
Chemical composition of clay used.

Formula	Mean percentage [wt%]
SiO_2	48.32%
TiO_2	0.83%
Al_2O_3	19.75%
Fe_2O_3	5.07%
MgO	2.30%
CaO	7.71%
Na_2O	0.79%
K_2O	2.93%
L.O.I.	16.08%

All test specimens were checked in order to find any fault as cracks or fissures. Flatness and orthogonally were also controlled by following the EN 772-16 (2011) and EN 772-20:2000/A1 (2005) standards. Specimens had a square shape of 400 mm × 400 mm. These dimensions were imposed by the equipment used to determine TC. Once the thermal assay was completed, specimens were random perforated with diamond cutter head with an interior diameter of 19 mm ±2%. These cylinders were gathered and used to determine the compressive strength (CS).

In all cases, each specimen was undergone a further drying process up to 110 °C to offset any rehydration before the test. Specimens were also measured in height and length by using a caliper.

3.5. Wall behavior simulation

Standards in each country regulate the thermal insulation capacity of the entire enclosure rather than the thermal conductivity of the material forming the ceramic block. Accordingly, this paper also examines the influence of the conductivity of the clay-waste mixture on heat transfer of single-leaf walls for a typical wall assembly.

The main target is to quantify the potential wall insulation enhancement achieved without manufacturing the bricks. This production implies a minimum load for the extruder of tons with the corresponding environmental impact, while the final product may have no commercial value. In order to minimize the energy and environmental impact, samples were made at laboratories facilities with the geometry required and after determining the physical behaviors a wall was simulated. To conduct the study, single-leaf walls with large format lightweight clay blocks were chosen, with a conventional assembly using standard mortar (λ_m = 1.3 W/m k), 10 mm thick tendel and furrowed-bed horizontal joint with a 30 mm air gap. Our manufacturer partner provided Termoarcilla® ECO2 brick geometry in order to design the wall.

The numerical calculation method (Morales et al., 2011a, 2011b) used to obtain the equivalent thermal transmittance (U_{eq}) [W/m² K] of a wall is in accordance with European and international standards: EN ISO 6946 (1996), EN ISO 10211-1 (2007) and EN ISO 10211-2 (2007).

Table 3
Chemical analysis of vine shoots.

Element	Mean percentage [wt%]
Ash	2.99%
Carbon	46.21%
Hydrogen	6.02%
Nitrogen	0.76%
Sulfur	0.06%
Chlorine	<0.01%
HHV	18,597.0 kJ/kg
Moisture	52.8%

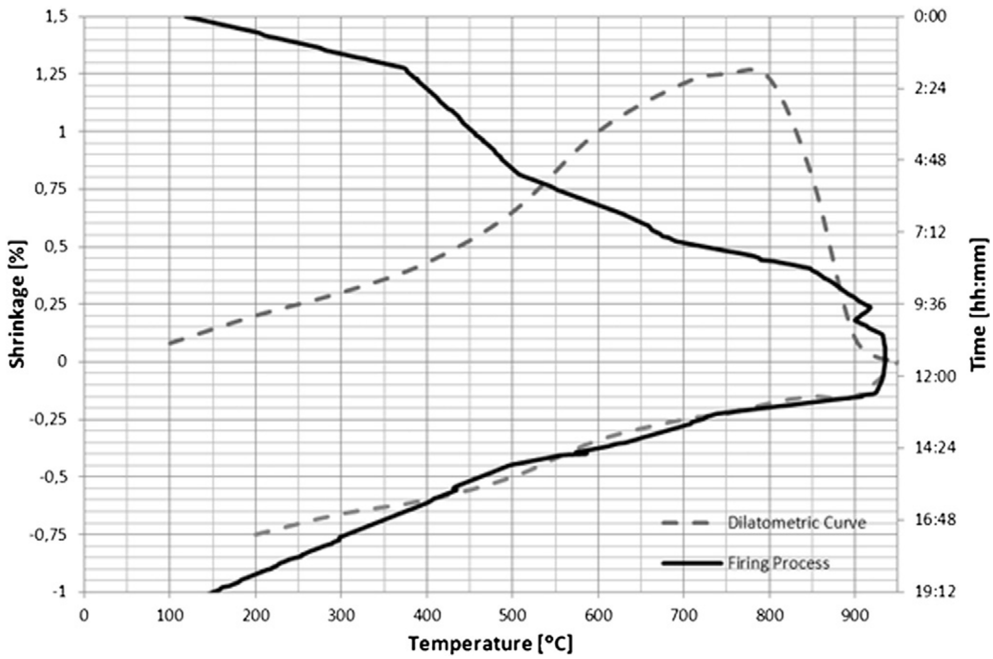

Fig. 2. Dilatometric curve and firing process followed.

4. Results and discussion

The elemental analysis of both, clay and kindling are shown in Tables 2 and 3 respectively. In addition, the dilatometric curve and its correlating firing cycle is shown in Fig. 2 where kiln tunnel temperature is the continuous line and dilatometric behavior the discontinuous ones.

As it is shown, the dilatometric curve shows an important expansion between 500 °C and 600 °C, which correspond to the transformation of quartz α into β. Another zone that must be highlighted is the one between 800 °C and 900 °C. Here samples show a fast shrinkage. In this area, the vitrification starts due to the action of K_2O as a fluxing agent. Then the next critical zone is in the cooling phase between 600 °C and 500 °C, where the free quartz can produce cracks due to the different shrinkage.

Based on this, it may be concluded that the waste will be incinerated before the first critical zone where the samples will arrive drier, and due to this, the risk of cracks is reduced. The firing curve has been adjusted to the above-mentioned behavior. In the first stage, a temperature of 400 °C is achieved in 2.5 h, and then the heating rate decreases up to 55 °C/h between 500 °C and 650 °C. The maximum temperature (950 °C) is reached after 2 h and samples remain there for 3 h. The first cooling stage, up to 700 °C, is very fast and take less than 1 h. The final stage takes 5 h and finish at 150 °C. At the end, samples did not show any crack or fissure.

The figures shown below were obtained with Statistica 8.0 software, where normality and variance were studied. Kruskal-Wallis tests (K-W) were performed to assess the significance of the observed results.

First, the HHV of kindling is shown in Fig. 3. Specimens weighted between 0.8 and 1.2 g. and the mean moisture was found around 52%.

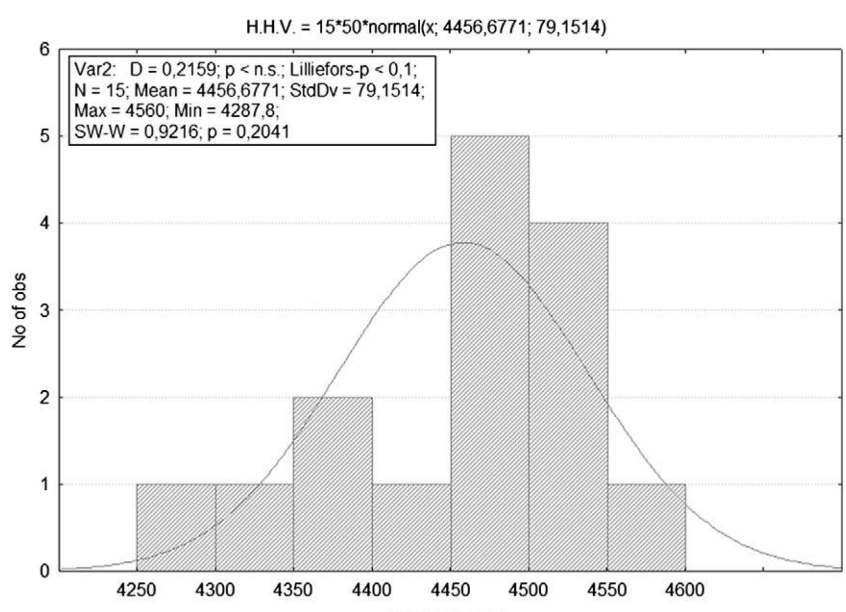

Fig. 3. HHV of waste.

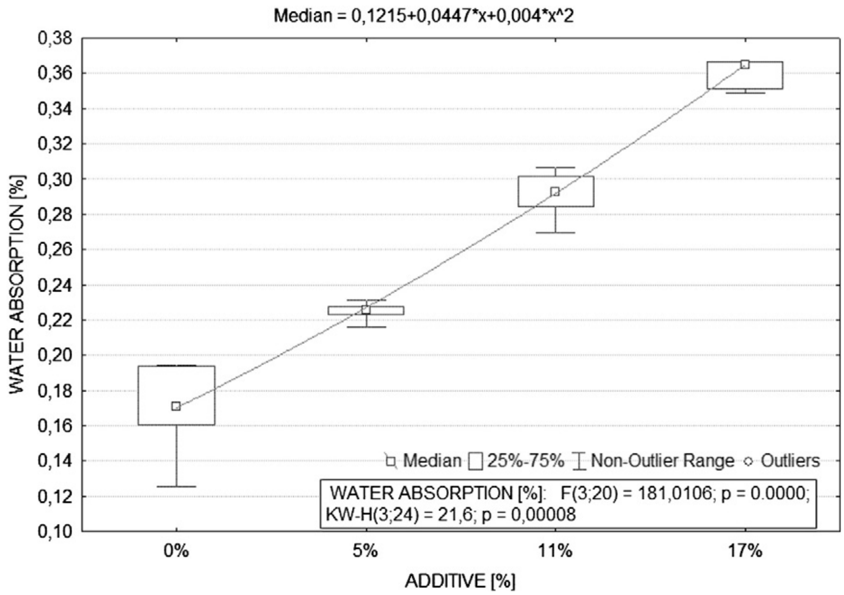

Fig. 4. Water absorption by percentage of waste.

The results obtained by the calorimeter are in accordance with previous researches (Demirbas, 1997).

Water absorption (Fig. 4) has ranged from 16.93% of AA00 to 36.04% of AS17. These results are in accordance with other previous (Demir, 2008; Bachir and Halima, 2012; Eliche-Quesada et al., 2012; Barbieri et al., 2013). Despite these references use sawdust instead of kindling, these showed the same trend and same decreasing percentage, for the same amount of waste added with a maximum particle size varying from 600 μm (Barbieri et al., 2013) to 1.6 mm (Bachir and Halima, 2012). This means WA is not highly influenced by the additive particle size. Water absorption depends on the volume of pores and its size but also on the connectivity between them (Cultrone et al., 2004). Connectivity is highly influenced by the firing temperature and the mineral composition and the reaction between minerals during the melting. The waste is burned before this melting point, and due to this, the initial particle size does not influence highly the pore formation process.

Thus, references report WA values that range from a minimum of 27% (Eliche-Quesada et al., 2012) to a maximum of 31% (Demir, 2008) when 10% of waste is added, while in this paper, for same percentage of waste, WA results 29.1%.

Values above 20% are not recommended for a durable performance of masonry (Daoud and Robert, 1992; Künzel and Kiessl, 1996; Camino et al., 2014). In case of water absorption exceeding 20%, these materials must be used by coating the outside facade, a common practice in building enclosures. In other cases, weathering exposition will reduce drastically the wall life cycle.

BD variation shows the same trend as the open porosity (see Fig. 5). This means there is high pore connectivity between the surface and the core of samples. This is clearly shown in Fig. 6 where correlation between BD and the open porosity is draw.

BD (Fig. 7) has decreased from 1,684 kg/m^3 to 1,124 kg/m^3, i.e., this addition causes a high weight reduction. Same trend and values were

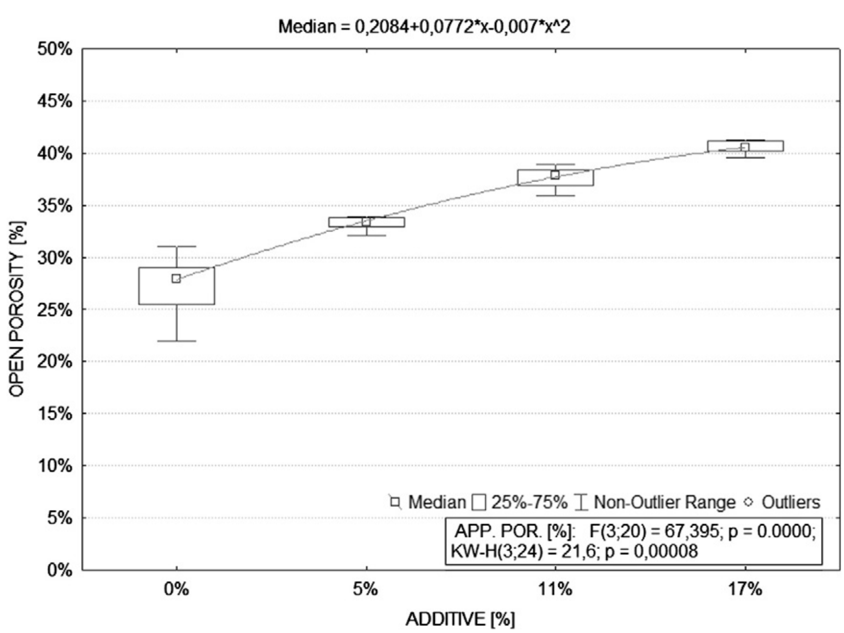

Fig. 5. Variation of open porosity by percentage of waste.

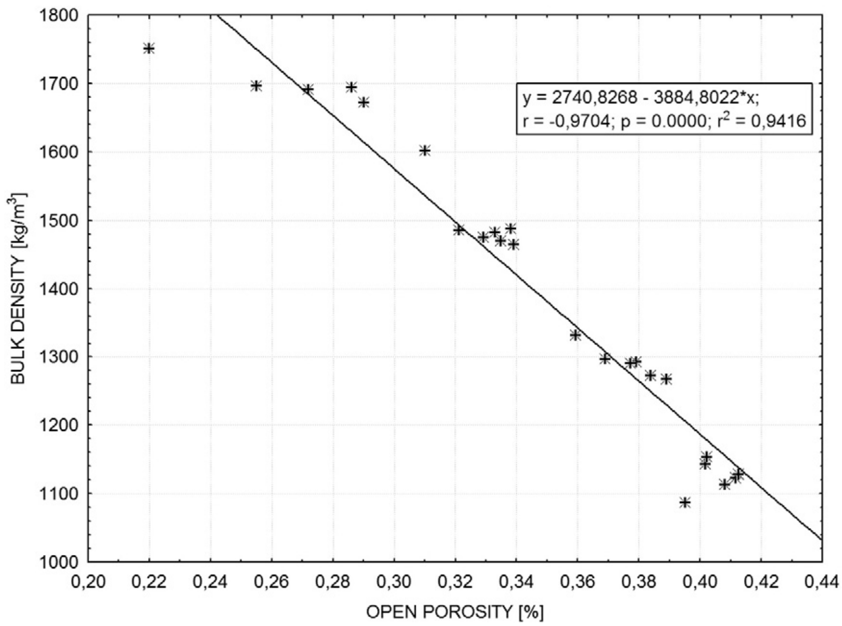

Fig. 6. Bulk density correlated with open porosity.

shown by previous researches which showed a BD decreasing of 13% (Bachir and Halima, 2012), 16% (Eliche-Quesada et al., 2012) and 25% (Demir, 2008), when 10% of waste is added. This property is a key factor for managers, taking into account the high economical costs related to logistic.

Linear shrinkage is also an important parameter for bricks manufacturers due to it influences on the extrusion die design. In order to obtain the brick desired geometry after the firing process, total linear shrinkage data are mandatory. In addition, linear shrinkage influences the risk of dimensional defects in bricks.

Previous researches have shown a high scattering value, from 2% to 6% (Demir, 2008; Bachir and Halima, 2012; Eliche-Quesada et al., 2012; Barbieri et al., 2013). In this paper, values are found also in this range, up to 10% of waste added. Fig. 8 shows the variation of LS from 6.34% of AA00 to 2.33% of AS17.

Thermal conductivity has been not reported by previous referenced researches (Demir, 2008; Bachir and Halima, 2012; Eliche-Quesada et al., 2012; Barbieri et al., 2013) concerning sawdust or kindling. Besides, to our knowledge, there is only one research that determines the thermal conductivity by the hot-wire method conducted by García-Ten et al. (2010) where lower reduction is shown (approx. 50%), for a maximum percentage of 11% of sawdust, while thermal conductivity in this paper decreases up to 60% for same percentage. Differences in firing temperature and mineral composition produce different porosity and, due to this, different thermal conductivity and mechanical behavior (Cultrone et al., 2004). In particular, it is shown that an increasing of CaO reduces significantly the thermal conductivity (García-Ten et al., 2005). CaO reacts at the calcite grain boundaries with Al_2O_3 and SiO_2 to form calcium crystalline phases and therefore the high porosity stemming from calcite particle decomposition leads to low

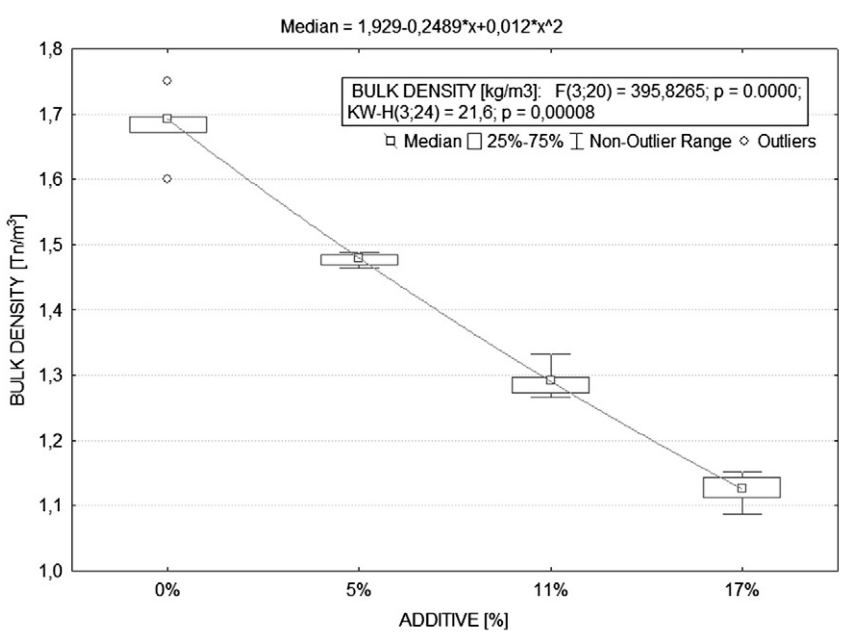

Fig. 7. Variation of bulk density by percentage of waste.

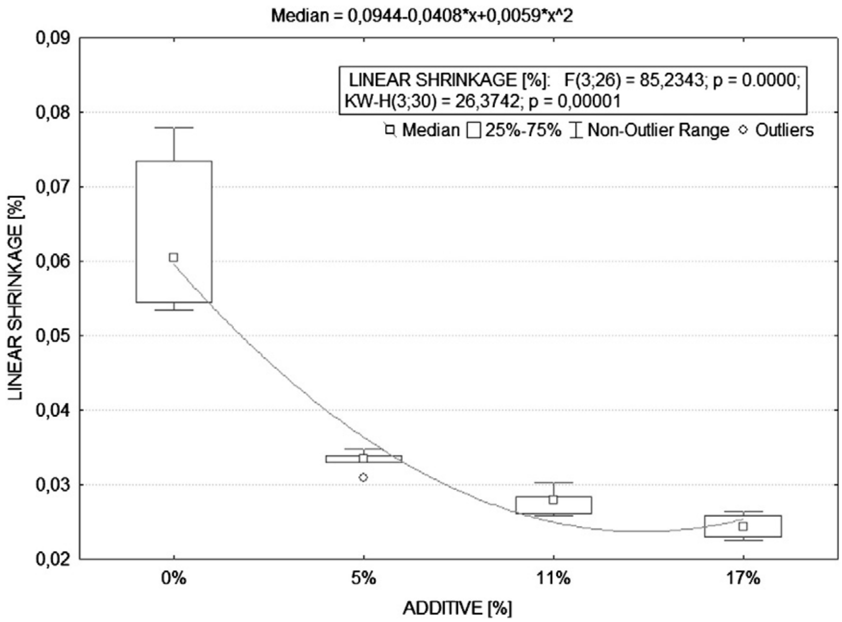

Fig. 8. Variation of linear shrinkage by percentage of waste.

thermal conductivity. The CaO content is increased in the matrix due to the ashes from kindling combustion. On one hand, this CaO increasing has also influence on the mechanical properties of the fired sample. Mechanical strength decreases because of increased fired porosity and microstructural flaw size. On the other hand, the quartz particles, from ashes, have much more negative impact than CaO due to this material provides large particles that do not fuse or are dissolved during firing. Then during cooling, the larger particle size and the polymorphic transformation of $\beta \rightarrow \alpha$ further reduce mechanical strength due to the different shrinkage of quartz grain what separates it from the matrix increasing the microstructural flaw size. Other previous researches have shown also an important reduction trend, finding around 50% and 70% reduction for 5% and 10% of waste added, respectively (Devant et al., 2011; Eliche-Quesada et al., 2012; Barbieri et al., 2013), while in this research the reduction is 76% for just 5% of waste. Since the firing temperature (950 °C) was the same as in this research and clay compositions were also similar to this, the difference may be understood if the mineral composition of ashes is considered. In the case of woody biomass ashes, CaO percentage (mean approx. 43.03%) is much greater than SiO_2 (mean approx. 22.22%). On the contrary ash from agriculture biomass shows more percentage of SiO_2 (mean approx. 33.39%) than CaO (mean approx. 14.86%) (Stanislav et al., 2013). Fired clay bricks are nowadays mostly used as part of the enclosure and rarely as a structural block, for supporting loads. Therefore, its thermal behavior has become the most important parameter, thus influencing wall thermal transmittance and energy consumption. TC at 10 °C (λ_{10}) (Fig. 9) and CS (Fig. 10) behavior have opposite trends. According to CTE DB SE-F (2012), CS should not be less than 5 N/mm^2 when bricks are not required for structural purposes. Specimens with concentration of waste up to 11% are above this limit. That is, TC of fired bricks can be reduced dramatically from 0.738 W/m K (clay without waste) to 0.280 W/m K (clay with 11% of vine shoot kindling).

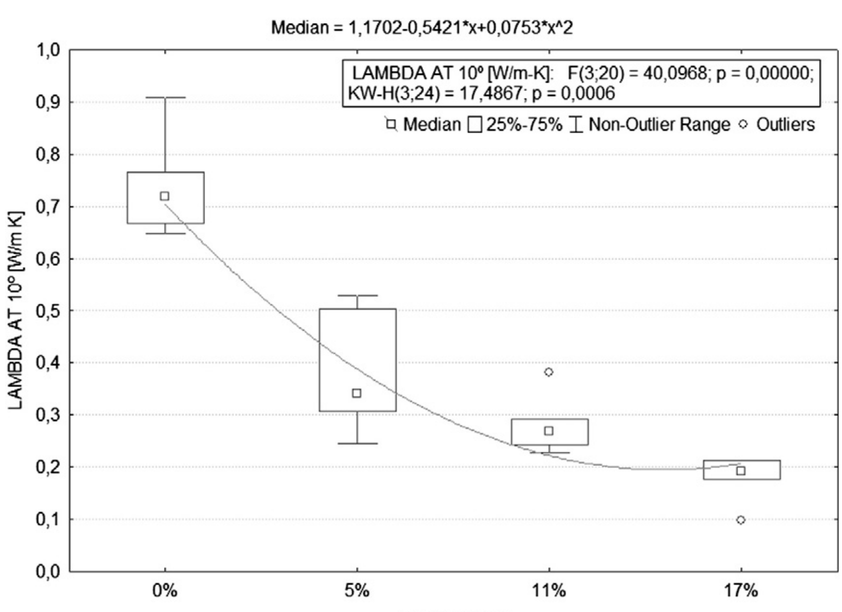

Fig. 9. Variation of thermal conductivity at 10 °C by percentage of waste.

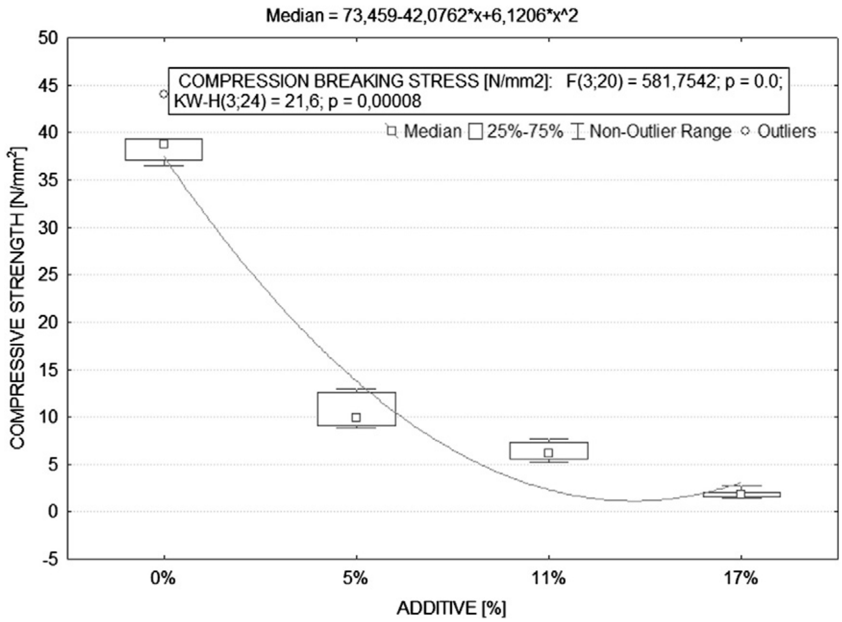

Fig. 10. Variation of compression strength by percentage of waste.

With the conductivity levels obtained for the various clay-waste mixtures, we sought to compare the equivalent thermal transmittance in the exterior wall of an actual facade. For clay without waste (with a thermal conductivity $\lambda_{10} = 0.738$ W/m k), the equivalent thermal transmittance of the wall was found to be $U_{eq} = 0.720$ W/m^2 K, and for clay with 11% of vine shoot particles added (with a thermal conductivity of $\lambda_{10} = 0.280$ W/m k), a $U_{eq} = 0.477$ W/m^2 K was determined. This represents a 34% improvement in the equivalent thermal transmittance of the wall. Table 4 resumes the mean values obtained related to the standard deviation.

Finally, it must be mentioned that the use of organic matters may impact on the exhaust gas emissions and on the facilities. Although chlorine (Cl), fluorine (F) and sulfur (S) clay contents have not been determined at this point, the next stage, for the process scaling up, should be taken into account this question. According to González et al. (2006) and Galán et al. (2002), the main possible sources for Cl are both phyllosilicates and halite and for S are pirite and gypsum. In the case of fluorine, same authors conclude that fluorine content ranges widely for similar theoretical composition and firing conditions, where the main sources in raw materials are illite, smectites and fluorapatite. Considering the firing temperature settled in this research (950 °C), the high content of CaO and the lack of efflorescence on fired samples, it is expected that F and S contained in the blend will react during the firing process producing CaF_2 and $CaSO_4$ and will therefore result in low F and S emissions. However, these emissions must be controlled by analyzing the exhaust gas in the mass production phase.

In the case of kindling, chlorine has been determined lower than 0.01% (see on Table 3). This small percentage, added to the one provided by the clay, mainly through the presence of halite, may generate harmful emissions that must be controlled. This situation was taken into account when the waste was selected. However, before installing the gas burners in the partner factory, the tunnel kiln was fed with biomass. It must be noted that the refractory material and the melting of Cl jointly N^+ and K^+ ions commonly interact, producing severe damage to the refractory material and due to this cracks or structural damages should be expected in the tunnel. However, such problems were not reported by the partner factory, while tunnel kiln was feeding with biomass. Although there is no expected worthy Cl emissions, before scale up, the waste brick production, an environmental analysis must be developed, as it has been mentioned previously.

5. Conclusions

The use of kindling from vine shoot as an additive for making fired clay bricks has been shown as another feasible new way for recycling vine shoots.

It is possible to decrease the thermal conductivity of fired clay bricks up to 62% by adding 11% of kindling from vine shoot, reaching a thermal conductivity value of 0.280 W/m k. If this percentage of 11% is not exceeded, bricks comply with the mandatory limit for the CS (minimum of 5 N/mm^2).

Therefore, a decreasing of equivalent thermal transmittance is expected for the masonry wall, assembled with such bricks. Indeed, the equivalent thermal transmittance of a wall can be decreased by up to 34% by reducing the conductivity of the clay by up to 62% without changing the block geometry or the type of wall assembly.

Fired clay bricks made by adding up to 11% kindling from vine shoot enhance thermal properties of bricks while water absorption exceeds

Table 4
Results summary.

PA [%]	AP [%]		λ_{10} [W/m K]		BD [kg/m^3]		CS [N/mm^2]		LS [%]		WA [%]	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
0%	27.22	3.14	0.738	0.095	1.684	0.049	38.041	4.49	6.34	1.01	16.93	2.50
5%	33.25	0.67	0.378	0.113	1.477	0.009	9.045	2.996	3.33	0.12	22.51	0.53
11%	37.63	1.06	0.280	0.056	1.292	0.023	6.043	1.560	2.76	0.15%	29.14	1.32
17%	40.52	0.67	0.208	0.091	1.124	0.023	1.556	0.615	2.33	0.40	36.04	0.80
K-W	21.6		17.487		21.6		21.6		26.37		21.6	
p	0.00008		0.0006		0.00008		0.00008		0.00001		0.00008	

recommended limit of 20%. Therefore, these bricks made of this mixture should be destined to form part of walls with coating, which constitutes a common practice in building enclosures.

At the same time, specimens had a uniform reddish color and did not show any efflorescence.

The combustion of vine shoot particles in the kiln involves savings in the fuel consumption during the firing process. Moreover, this energy provided by the waste is considered having a net zero carbon footprint since it is bio-fuel. However, the waste chemical composition may produce during the firing process harmful emissions that negatively impact on both the environment and the factory facilities. For this reason, before scaling up the mass production, a deep analysis of the Cl, S and F gases must be carried out.

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