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Fly ash addition in clayey materials to improve the quality of solid bricks

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The technical quality of two compositionally different groups of solid bricks fired between 800 and 1000 °C was evaluated. Five weight percentage of fly ash was added to both groups and they were compared with similar bricks with no added fly ash.

The textures of the bricks with fly ash were very similar to the textures of those without it, except that the samples with the additive contained spherical fly ash particles with diameters ranging from 0.1 to 10 μm. These particles led to a reduction in the density of the bricks and a substantial improvement in their durability, with less decay being caused by salt crystallization in the pores. This is because fly ash causes a reduction in the number of micropores, the pores that make porous materials most vulnerable to salt-induced decay. Use of this additive could have practical implications as a means of recycling and for achieving cost savings in brick production.

1. Introduction

A lot of research is currently being done into recycling, into how to reuse the waste we produce in our daily lives.

The building industry has always shown a receptive attitude to research into new materials [1,2]. Fly ash, a by-product of coal combustion, is frequently used in concrete production as an inexpensive substitute for Portland cement. Its pozzolanic properties improve the strength of the concrete, and its small particles make the mixture easier to knead [3].

Indeed, there is an extensive bibliography on the use of fly ash as a component of concrete [4,5] and the changes that its addition induces in both mechanical [6] and thermal [7] terms. In the brick-making industry, there has also been research into how to reuse different waste products in order to manufacture better quality bricks [8–11]. However, although fly ash is commonly used in cements, it has rarely been applied to bricks. Recently it has been shown that fly ash might improve the compressive strength of bricks and make them more resistant to frost [12]. Cicek and Tanriverdi [13] observed the positive effect of the addition of fly ash, sand and hydrated lime in the compressive strength of the bricks. In the field of Architectural Heritage, there is almost no research about fly ash addition in the manufacture of replacement bricks for use in restoration work. An essential criterion in this case is that the bricks must have the same physical and mechanical parameters (colour, porosity, hydraulic properties, etc.) as the original bricks used in the building being restored.

Brick is an extremely old building material, known to have been used in the Mesopotamia region since the third millennium BC [14]. The term "brick" encompasses a wide number of products obtained by mixing clay, preparing and moulding it, before slow drying and finally firing in an oven. As the temperature rises, mineralogical and textural changes occur. These are the result of the marked disequilibrium of a system that on a small scale resembles high-temperature metamorphic processes [15]. The porosity of the brick depends directly on the mineralogical composition of the raw material and the firing temperature, but generally, bricks fired at high temperatures are more vitreous and undergo the greatest changes in size and porosity [16].

The aim of this work is to evaluate the technical quality of solid bricks with added fly ash for use in building restoration. The sample bricks were made using two types of clayey raw materials and were then fired at different temperatures. The physical and mechanical properties and the durability of the bricks were then compared to those of similar bricks with no added fly ash, so as to assess whether the quality of the bricks had been enhanced or at least maintained. The use of this additive in bricks could produce cost savings in raw materials for brick manufacturers and serve as an efficient means of recycling a waste product.

2. Materials and methods

2.1. Samples

Solid bricks were prepared using two different raw materials (one from Guadix and the other from Viznar) used in Granada (Andalusia, Spain) since ancient times for the manufacture of ceramic products. The clayey material from Viznar (V) contains

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significant amounts of carbonate minerals (10% of calcite and dolomite), whereas the material from Guadix (G) is mainly quartz and has no carbonates [15]. According to Shepard's diagram [17], both raw materials can be classified as silty-sand materials. They are rich in SiO_2 and, only in Viznar, CaO + MgO reach 13 wt.% (Table 1). Bricks were fired in an air-ventilated electric oven (Herotec CR-35) at 800, 900 and 1000 °C, the temperatures most frequently used in the brick industry.

The fly ash came from the electrofilter of the "Los Barrios" coal-fired power station in Seville (Andalusia, Spain). Particle size ranged from 0.1 to 10 μm (Fig. 1) and its chemical composition is shown in Table 1. The SiO_2 and Al_2O_3 contents make up 76.4 wt.% of the fly ash sample and CaO accounts for 9.2 wt.%. The real density and the specific surface area are 2.31 g/cm^3 and 0.45 m^2/g, respectively [18].

The addition of large amounts of fly ash can cause colour changes in the bricks, although their quality can remain unaffected. As our aim was to produce new bricks that could be used in restoration work alongside the original pieces it was important to maintain the same colour. We therefore used colorimetry to determine the maximum amount of fly ash that could be added to the bricks without significantly affecting the colour.

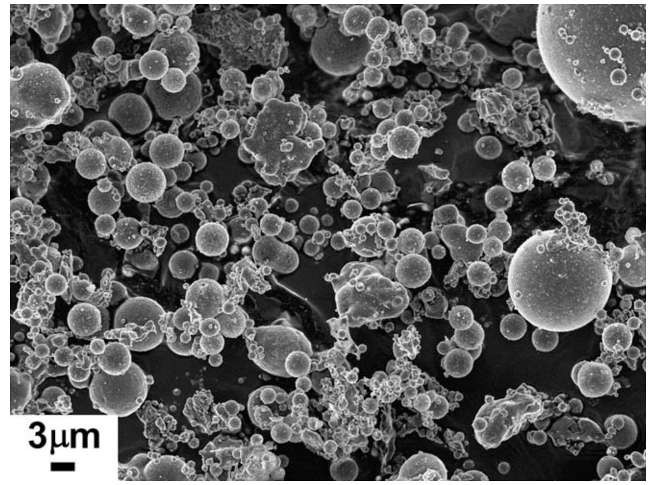

Fig. 1. FESEM microphotography showing the morphology of fly ash particles.

2.2. Analyses

To determine the amount (wt.%) of fly ash to be added to the bricks, colour measurements were performed using a portable Minolta CR 210 colorimeter with a 0° view angle and 50 mm diameter measuring area. Three measurements per sample were carried out by selecting CIE illuminant C (simulating daylight with a colour temperature of 6774 K) on Viznar and Guadix samples fired at 900 °C and with an increasing percentage of additive (0.5, 1, 2, 5, 10 and 15 wt.%). Colour data were obtained using $L^*a^*b^*$ coordinates.

Once we had determined the maximum amount of fly ash that could be added without significantly changing the colour of the bricks, we then evaluated the durability and petrophysical properties of the samples.

The texture of the bricks was studied using Leo Gemini 1530 field emission scanning electron microscopy (FESEM) coupled with Oxford INCA 200 microanalysis. FESEM secondary electron (SE) images were acquired using small carbon coated sample pieces (5 × 5 × 3 mm in size).

The parameters associated with the uptake of fluid and its movement inside the pores were determined by hydric tests. Since decay processes often depend on the circulation of water inside porous solids [19], these tests were important for determining the durability of these solids. Water absorption [20] and drying [21] were measured by weighing the samples (three samples per group) at regular intervals. The values obtained were then used to calculate the absorption and saturation coefficients, the real and apparent density and the open porosity.

The distribution of the pore access size and the pore volume were measured using a Micromeritics Autopore III 9410 porosimeter. Two cubic centimeter sample chips were oven dried for 24 h at 110 °C and then analysed. Two MIP measurements were made per sample.

There are various techniques for determining the physical properties of the bricks, of which ultrasound procedures are perhaps the most appropriate in this case because of their non-destructive nature. The measurements were performed with a Panametrics HV Pulser/Receiver 5058PR coupled with a Tektronix TDS 3012B oscilloscope. The propagation velocity of compressional (V_P) and shear (V_S) pulses was measured in accordance with the ASTM D 2845 [22] standard for dry test samples using polarized Panametric transducers of 1 GHz. These data were used to obtain information on the degree of compactness of the bricks. We measured the velocity of the ultrasound waves under controlled thermo-hygrometric conditions (~25 °C and relative humidity of ~50%). A visco-elastic couplant was used to ensure good coupling between transducers and brick samples. The transmission method was used and three measurements were taken for each direction under consideration. Once we had established the wave velocity (V_P and V_S) and the brick density (ρ), it was possible to calculate the Poisson Coefficient (ν, Eq. (1)), Young (E, Eq. (2)), shear (G, Eq. (3)) and bulk (K, Eq. (4)) modules, which are useful when predicting the deformation behaviour of building materials [23,24]:

$$\nu = \frac{(V_P/V_S)^2 - 2}{2[(V_P/V_S)^2 - 1]} \quad (1)$$

$$E = \rho V_S^2 (1 + \nu) \quad (2)$$

$$G = \frac{E}{2(1 + \nu)} \quad (3)$$

$$K = \frac{E}{3(1 - 2\nu)} \quad (4)$$

A uniaxial compression test provided information on the behaviour of bricks subjected to mechanical forces and their capacity to resist stresses. Three measurements per sample were performed on 3 cm edge dry brick cubes using a Metro Com MI-30 press according to the UNI EN1926 standard [25].

Finally, we performed 10 salt crystallization cycles in accordance with the UNI EN 12370 [26] standard using a solution of

Table 1
Mayor elements (in wt.%) of Viznar (*V*) and Guadix (*G*) raw materials and of fly ash (fa) from "Los Barrios" coal-fired power station in Seville (Andalusia, Spain)

	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	PC
V	48.73	14.90	5.64	0.08	3.30	9.92	0.74	2.63	0.79	0.14	12.99
G	61.95	19.76	7.37	0.10	0.77	0.57	0.90	2.85	1.05	0.17	5.15
fa	43.42	32.98	2.41	0.05	2.09	9.20	0.14	0.49	1.66	1.56	5.03

14% $NaSO_4 \cdot 10H_2O$ to evaluate the durability of the brick. This test showed the damaging effects of soluble salts that are usually contained in water and can crystallize in the pores and fissures of bricks. The damage was assessed by means of a visual inspection of material loss and by measuring weight changes.

3. Results

3.1. Colorimetry

In both groups of bricks there was evidence of a progressive increase in lightness as the amount of fly ash was augmented (L^*, Table 2). Guadix samples needed at least 15 wt.% of fly ash to produce significant changes in colour, while in Viznar samples this happened when only 10 wt.% was added. Since one of the objectives of this research was to maintain the same colour as the bricks without additive, the amount of fly ash added to raw materials colorimetrically similar to those from Viznar and Guadix must be less than 10 wt.%. We therefore decided to add 5 wt.% of fly ash to the clayey materials.

3.2. SEM observations

Bricks fired at 800 °C still maintain the sheet-like appearance of phyllosilicates (Fig. 2a), although there is a noticeable mica (muscovite) exfoliation along their basal planes due to dehydroxylation [27]. Interconnection between particles is limited. At this temperature there is no clear evidence of vitrification or partial melting and the spherical fly ash particles are easily recognizable (Fig. 2b). Vitrification is evident when samples are fired between 900 and 1000 °C because smooth surfaces and ellipsoidal pores can be identified ("cellular structure", according to Tite and Maniatis [28]). This is due to the total or partial melting of clayey particles associated with the escape of trapped gases in the matrix of the samples [29]. The fly ash particles however do not melt even at temperatures as high as 1000 °C (Fig. 2c). Apart from this the texture of the bricks with and without fly ash is the same. Vitrification effects are less evident in samples with a high carbonate content (Viznar, Fig. 2d) than in samples rich in silicates (Guadix). This is due to the fact that when calcite and dolomite break down into CaO and MgO, the Ca and Mg are incorporated into the structure of new mineral phases, principally in high-temperature silicates such as gehlenite, wollastonite and diopside, which tend to obstruct the melting process [30].

3.3. Hydric properties

Prolonged retention of humidity inside building materials favours physicochemical decay processes. Bricks are porous materials and are therefore vulnerable to water-induced decay (when it rains for example). In this case the best materials are those that absorb water slowly and then dry out quickly [31]. Guadix bricks systematically absorb less water (A_b and A_f, Table 3) and their absorption velocity (C_a) falls as the firing temperature increases. The drying index demonstrates that the samples that shed water most quickly are those fired at 1000 °C (D_i, Table 3).

The Viznar samples do not behave in the same way as the Guadix samples when the firing temperature changes. In fact, SEM observations show that these bricks maintain the same texture when they are fired at different temperatures between 800 and 1000 °C. This is due to the fact that there is almost no melting process, something which is reflected in the unchanging values of A_b, A_f and C_a. The drying index (D_i, Table 3) for the samples fired at 1000 °C (*V*10) is higher than for those fired at 800 °C (*V*8) and 900 °C (*V*9). However, it is lower than the index for all the Guadix samples, whatever the firing temperature. Viznar bricks stand out for their high saturation coefficient values (*S*, Table 3). They also have higher open porosity values (P_o). When 5 wt.% of fly ash was added, the brick density was significantly lower (ρ_a and ρ_r, Table 3), because of the lower density of the additive (2.3 g/cm^3) compared to that of bricks (2.6–2.7 g/cm^3). The addition of fly ash did not change any other hydric parameters: V_{fa} and G_{fa} absorbed more water than *V* and *G* but their absorption (C_a) and drying (D_i) velocities remained almost the same. Therefore, although V_{fa} and G_{fa} absorb more water, they dry out as quickly as bricks without fly ash, which indicates that they are of similar quality under hydric tests. It is important to note that the hydric parameters shown in Table 3 are within the limits of standard values measured on brick masonries. For example, modern solid bricks are generally characterized by a ρ_a of $\sim$1.75 g/cm^3 and an A_b of less than 35% [31].

3.4. MIP measurements

Porometric curves, open porosity (*P*) and surface area (*A*) values are shown in Fig. 3. Pores with a 1 μm access radius are predominant in all the samples. However, only in Guadix samples does the pore size increase as the temperature rises. This result indirectly confirms the progressive vitrification of Guadix bricks, and the formation of bigger pores already visualized with SEM ("cellular structure", Fig. 2c). On the contrary, in Viznar, as the texture does not change (there is no melting), the pore size distribution is the same across the entire temperature range used in this study. It is important to note that the addition of fly ash leads to the decrease of the surface area (*A*, Fig. 3) in all bricks. This suggests that even the tiniest amount of fly ash reduces the proportion of the smallest pores. These pores are particularly dangerous for the durability of porous solid materials as, among other things, they help to augment the pressure caused by the crystallization of soluble salts in the pore walls [32].

The open porosity values obtained using this technique (*P*, Fig. 3) are always below the values calculated using the water absorption method (P_o, Table 3). Whiteley et al. [33] observed this discrepancy when analysing other building materials. The explanation lies in the fact that the analytical techniques are different and involve liquids with very different physical properties. Esbert et al. [31] pointed out that porosity values for bricks can vary greatly (15–40%) depending on the age of the brick and the manufacturing process, with the old bricks used in historical buildings being the most porous. Within this range the bricks we used can be classified as moderate to highly porous.

3.5. Physical–mechanical tests

Viznar and Guadix bricks with fly ash show a dynamic behaviour similar to that of the samples without additive (Table 4).

Table 2
Lightness (L^*) and chromatic coordinates (a^*yb^*) calculated for Viznar and Guadix samples fired at 900 °C with varying concentrations of fly ash (wt.%)

wt.%	Viznar			Guadix		
	L^*	a^*	b^*	L^*	a^*	b^*
0	56.75	14.15	21.89	55.59	19.29	27.97
0.5	57.16	13.49	21.58	56.76	20.23	28.92
1	57.62	13.73	22.03	56.81	19.98	28.75
2	59.26	13.82	21.96	56.36	20.10	28.45
5	60.52	13.96	22.12	56.23	20.55	28.69
10	62.45	12.37	19.89	56.09	20.53	28.83
15	65.20	10.75	19.05	58.86	18.10	24.36

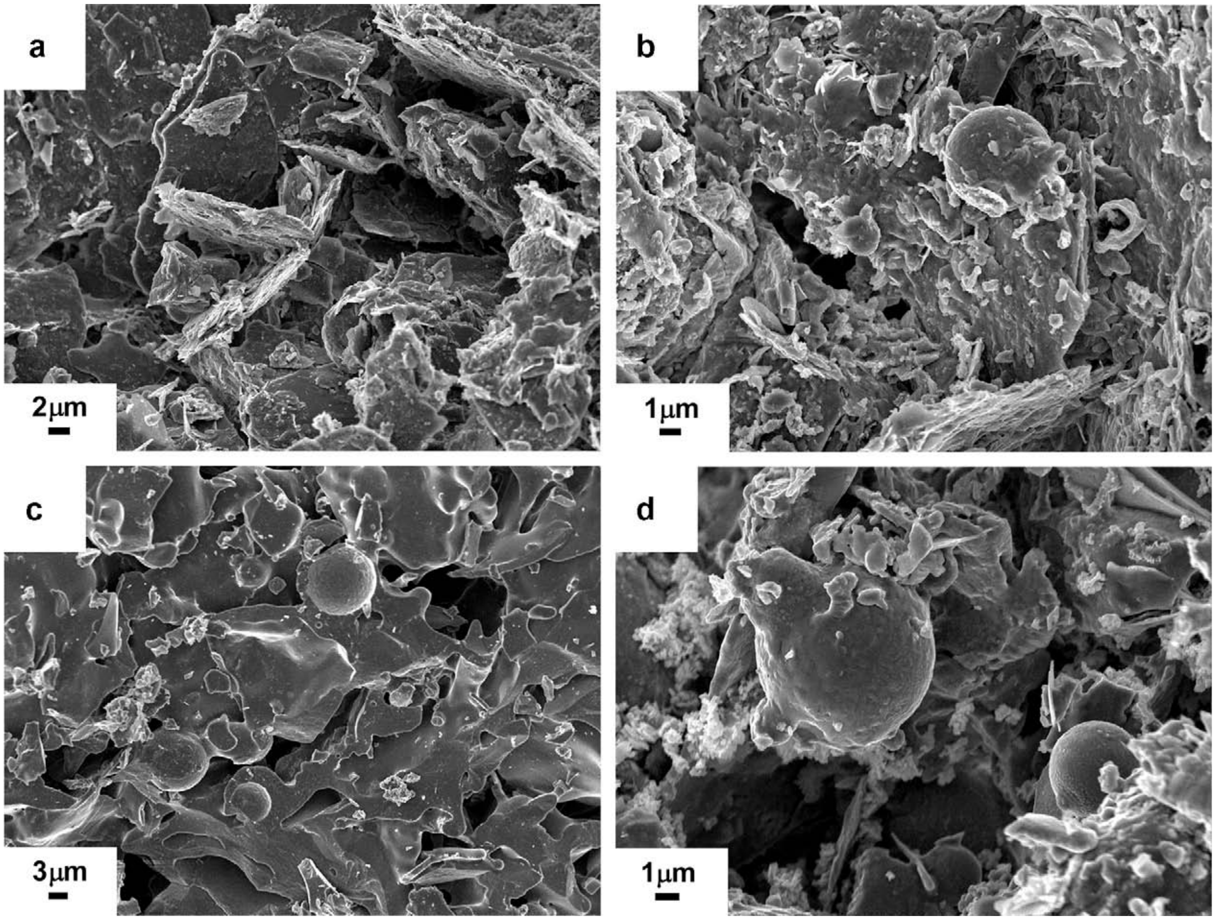

Fig. 2. FESEM microphotographs of different bricks: (a) Guadix without fly ash and fired at 800 °C; (b) Viznar with fly ash and fired at 800 °C; (c) Guadix with fly ash and fired at 1000 °C; (d) Viznar with fly ash and fired at 1000 °C.

Table 3
Hydric parameters for Viznar and Guadix bricks without additive (*V* and *G*) and with fly ash (V_{fa} and G_{fa}) fired at 800 (8), 900 (9) and 1000 °C (10)

	*V*8	*V*9	*V*10	V_{fa}8	V_{fa}9	V_{fa}10	*G*8	*G*9	*G*10	G_{fa}8	G_{fa}9	G_{fa}10
A_b (%)	25.58	23.69	25.39	24.38	27.08	28.56	24.84	23.15	14.94	27.17	26.22	21.00
A_f (%)	25.60	23.71	25.49	24.80	27.59	29.43	25.14	23.90	16.76	28.58	27.69	22.57
C_a	0.11	0.10	0.10	0.09	0.11	0.12	0.10	0.08	0.05	0.11	0.11	0.09
D_i	0.33	0.33	0.24	0.29	0.24	0.18	0.08	0.06	0.05	0.11	0.09	0.09
S (%)	95.49	92.45	91.07	89.62	89.90	87.96	88.16	82.96	74.98	86.82	85.53	82.76
P_o (%)	41.06	36.97	40.58	38.50	41.31	43.82	39.28	37.41	27.49	42.30	41.73	37.29
ρ_a (g cm^{-3})	1.60	1.60	1.60	1.55	1.50	1.42	1.58	1.61	1.83	1.48	1.51	1.65
ρ_r (g cm^{-3})	2.72	2.58	2.69	2.53	2.55	2.65	2.62	2.62	2.65	2.57	2.59	2.64

A_b = free absorption; A_f = forced absorption; C_a = absorption coefficient; D_i = drying index; *S* = saturation coefficient; P_o = open porosity; ρ_a = apparent density; ρ_r = real density.

V_P and V_S velocities in Viznar samples are higher than those for the Guadix samples when temperatures are low, and they are equal to or less than them when the temperature reaches 1000 °C. This is the consequence of the greater vitrification that Guadix samples suffer [15]. The presence of fly ash reduces the velocity of ultrasonic wave propagation, especially in Viznar samples. These results are to be expected given the lower density of fly ash bricks (ρ_a and ρ_r, Table 3).

The modules we calculated are controlled by the brick composition and the development of vitrification. In fact, Young modulus (*E*, Table 4) is higher in *V* than V_{fa} whilst *G* and G_{fa} show similar values: when the firing temperature is increased, the *E* value also rises particularly at 1000 °C, when vitrification is very widespread. These differences still remain when the bulk (*K*) and share (*G*) modulus are considered.

As regards the capacity of bricks to resist stresses, it is evident that the presence of fly ash reduces their resistance to uniaxial compressive forces (*R* values, Table 4). However, except for two cases (*G* and G_{fa} fired at 800 °C), the results for bricks with fly ash fall within the range suggested by the NBE-FL-90 standard [34] for masonry brick structures. The two samples outside the range could still be useful for restoration work, as old bricks can also show lower values than those measured in this work (1.5 MPa [31]).

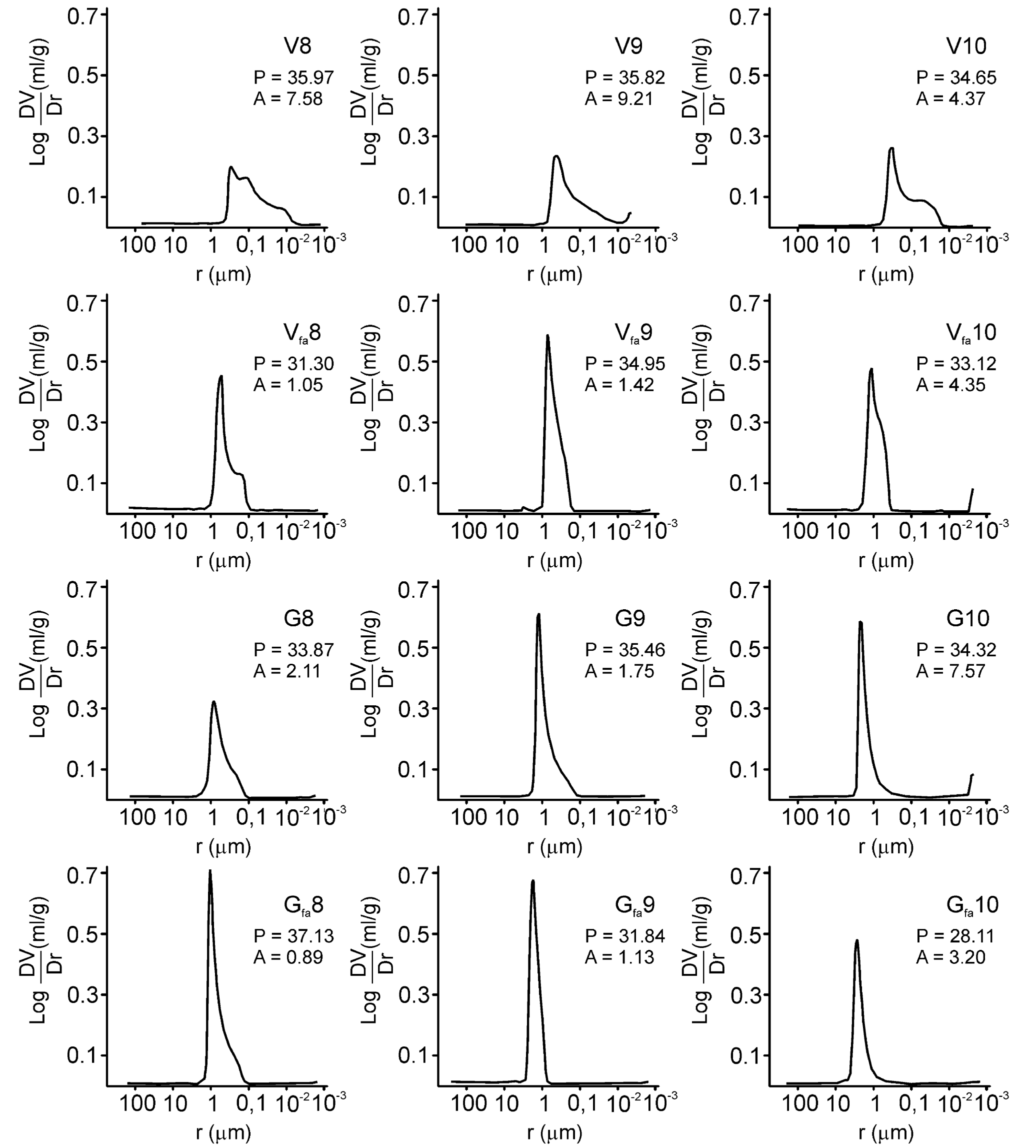

Fig. 3. MIP pore size distribution curves (i.e. log differential intruded volume (mg/l) versus pore radius (μm)) of Viznar and Guadix samples without (*V* and *G*) and with fly ash (V_{fa} and G_{fa}). The porosity (*P* in %) and superficial area (*A* in m^2/g) values are reported in each diagram.

3.6. Salt crystallization

The salt crystallization test showed that fly ash addition had highly beneficial effects in that it increased the durability of the bricks. In fact, when the values for the samples without fly ash in Fig. 4 (on the left) are compared with those for the samples with the additive (on the right), it is evident that weight change ($\Delta M/M$) in the latter was minimal through the 10 test cycles; especially if we take into account that the V_{fa} and G_{fa} scale is 1/10 of the *V* and *G* axis scale.

As far as the type of raw material is concerned, the Guadix samples that suffer the smallest weight variations as a consequence of sodium sulphate crystallization in their pores are those fired at 1000 °C, while those fired at lower temperatures show more damage with considerable fragment loss. This behaviour is due to the different vitrification level reached by these bricks. The fact that there were almost no changes in the texture of Viznar bricks fired between 800 and 1000 °C means that the graphs are very similar (Fig. 4b). In general, the response to this aging test was better in the samples from Viznar than in those from Guadix.

Table 4
Average velocities for the propagation of ultrasonic V_P and V_S pulses (m/s) and resistance values after uniaxial compression test

	T (°C)	V_P	V_S	*v*	*E* (×10³)	*G* (×10³)	*K* (×10³)	*R*
V	800	3252	1884	0.25	14.17	5.68	9.35	11.98
	900	3145	1813	0.25	13.16	5.26	8.81	13.39
	1000	3206	1718	0.30	12.26	4.72	10.15	13.00
V_{fa}	800	2434	1541	0.17	8.58	3.68	4.28	6.74
	900	2362	1450	0.20	7.55	3.15	4.16	5.93
	1000	2195	1379	0.17	6.34	2.70	3.24	8.02
G	800	1498	804	0.30	2.65	1.02	2.18	2.25
	900	1924	1206	0.18	5.51	2.34	2.84	9.00
	1000	3202	1855	0.25	15.71	6.30	10.37	15.33
G_{fa}	800	1273	780	0.20	2.16	0.90	1.20	2.30
	900	1587	924	0.24	3.21	1.29	2.08	5.19
	1000	3061	1824	0.22	13.45	5.49	8.14	5.56

Values for Poisson Coefficient (v), Young modulus *E* (in GPa), shear modulus G (in GPa), bulk modulus K (in GPa) and uniaxial compressive strength R (in MPa) in brick samples without added fly ash (V and G) and with added fly ash (V_{fa} and G_{fa}) fired at 800, 900 and 1000 °C.

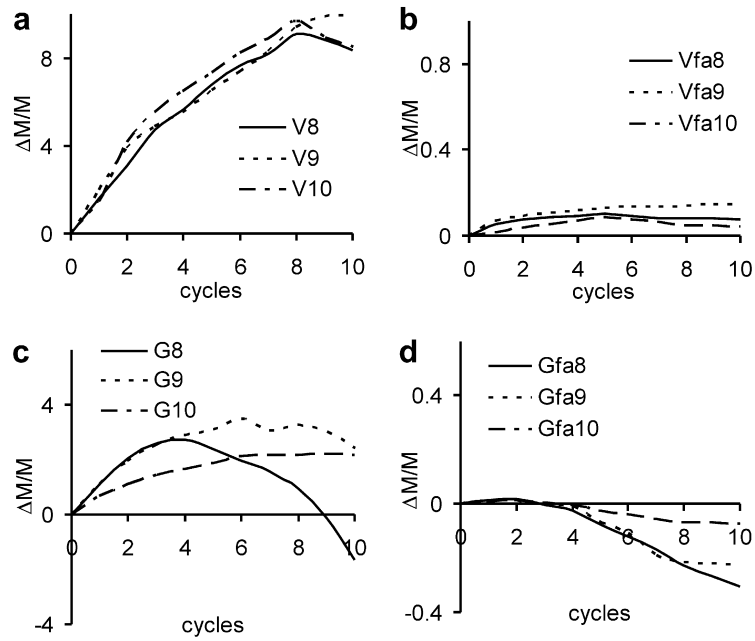

Fig. 4. Brick weight variation fired at 800 °C (8), 900 °C (9) and 1000 °C (10) during salt crystallization test: (a) Viznar without fly ash (*V*); (b) Viznar with fly ash (V_{fa}); (c) Guadix without fly ash (*G*); (d) Guadix with fly ash (G_{fa}).

4. Conclusions

After characterizing the texture and the petrophysical properties of handmade bricks with added fly ash, and evaluating their durability, the following conclusions can be drawn:

(1) As colour is one of the parameters to take into account when replacing damaged bricks in historical buildings, no more than 10 wt.% fly ash should be added for the types of raw material used in this work. Higher quantities would lead to significant changes in chromatism and lightness, when compared with the original bricks in the building being restored.
(2) From a textural point of view the bricks do not undergo any obvious changes except for the presence of spherical fly ash particles with diameters ranging from 0.1 to 10 μm. These particles are dispersed in a clayey matrix with a greater or lesser degree of vitrification depending on the type of raw material used (with or without carbonates) and the firing temperature (800, 900 or 1000 °C).
(3) Fly ash does not modify the hydric properties of the bricks but it does make them lighter. In fact, all the bricks with fly ash have a lower density.
(4) Dynamo-elastic properties are similar in all cases, although ultrasound velocities are slightly lower in bricks with fly ash, as they have a lower density.
(5) Fly ash bricks show less damage than conventional bricks when exposed to salt crystallization cycles. This improvement is due to the reduction of the surface area of the bricks, i.e. the reduction of the volume of the smallest pores, the ones that cause most damage to the bricks due to soluble salt crystallization.

These results show that the addition of fly ash can enhance the quality of the brick, although for restoration purposes if too much fly ash (⩾10 wt.%) is added, this can spoil the aesthetic appearance of the buildings being restored, due to excessive colour differences. Bricks with larger amounts of fly ash could however be considered for use in the construction of new buildings, but only after they have been subject to a detailed petrophysical characterization study. It is also important to point out that the raw materials used in this work are very common in terms of their mineralogy. This means that this research has a general application for a large number of brick-making companies who could consider using fly ash in their production process. This would encourage the recycling of this waste product and help to reduce manufacturing costs, as fewer raw materials would be required.

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