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Ultrasound and mechanical tests combined with ANOVA to evaluate brick quality

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Abstract

Mixtures of clays are often used in the manufacture of bricks, with distinct additives for diverse ends. The firing process, however, determines the final properties of the material. In this work, ultrasound and mechanical tests have been combined with a statistical tool, the analysis of variance (ANOVA), to analyse the mineralogical and physical characteristics of solid bricks manufactured from mixtures of local clays to which small amounts of additives have been included. They were then fired at different temperatures, ranging from 850 up to 1100°C. A new parameter, defined as the "resistance anisotropy" has been formulated to simplify the statistical interpretations. In addition, a two-way ANOVA interaction analysis has been used to evaluate the effect that selected factors (addition/absence of additives and firing temperature) have on the technical quality of bricks. © 2001 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

Keywords: C. Mechanical properties; Ultrasonic testing; Resistance anisotropy; ANOVA; Technical quality of bricks

1. Introduction

Bricks are an extremely old building material, known to have been used in constructions since before 1530 B.C. in Egypt [1]. Currently, bricks have been found to be highly deteriorated in many constructions considered part of our architectural heritage.

The term "brick" encompasses a wide number of products obtained by mixing clay, preparing and moulding it, then submitting it to slow drying and finally firing in appropriate kilns. Although the distinct additives that can be used have very diverse ends [2], a temper is usually added to the clay to reduce its shrinkage during drying and firing. This can affect the behaviour of the brick when carbonated materials (calcite or dolomite) are used as tempers, since lime blowing is then common [3,4]. On the other hand, the particle size used influences the resistance of bricks. Generally, an increase in particle size causes a reduction in the plastic contraction during the processing conditions, but decreases the deformational stress [5].

The plasticity of the clay, the firing heat and the porosity of the brick are three of the most fundamental properties characterising bricks. Analytical techniques such as XRF, XRD, optical microscopy, porosimetry, and mechanical tests, among others, are frequently used to examine these properties. The recent incorporation to this list of ultrasound tests has allowed the non-destructive study of stone, lime mortars [6], and bricks [7]. Ultrasound tests are based on the introduction of longitudinal (V_P) ultrasonic waves (from 2×10^4–10^3 Hz) by periodic pulses using a transducer (emitter) coupled to the surface. After travelling through the brick, the waves are collected by another transducer (receiver). The changes in the waves are then measured, as well as the time elapsed between emission and reception [$V_p = d/t$, where d is the distance covered in cm (measured by callipers) and t is the time in μs for the wave to reach the receptor]. The velocity, due to the heterogeneous, discontinuous and anisotropic nature of the bricks, is influenced by their mineralogical composition, intercrystalline connections, density, humidity, and the

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presence of hollows, fissures or cracks, reflected by a drop in the velocity as a result of absorption and dispersion [8,9]. As the study of the technical quality and/or the state of preservation of a construction material should preferentially be based on the use of NDT, techniques based on the propagation of ultrasound waves are obviously advantageous.

Mechanical tests (uniaxial compression tests) provide information on the behaviour of bricks subjected to mechanical forces and their capacity for resisting stresses. These tests are of great use since, above a certain pressure, samples can undergo irreversible physical damage, contributing to the deterioration of the material and even to its structural instability.

The aim of the present study was to examine the technical behaviour of bricks in order to determine the effects that the addition/absence of additives, and firing temperature produce on the brick quality. To do so, the two tests described above, ultrasound and mechanical, were used to obtain measurements. To simplify the statistical interpretation, a new parameter defined as the "resistance anisotropy" has been established, calculated on the results obtained by the other two techniques. Variance Analysis (ANOVA) [10] has been applied to obtain more information with respect to the similarities and differences in the behaviour of the samples to these techniques.

2. Experimental procedures

Solid bricks (10×7×3 cm) were prepared in accordance with Regulation NB-FL-90 [11] using a mixture of pliocenic clays from detritic deposits in Guadix (Granada, Spain) [12] and silty deposits in La Gabia (Granada, Spain). The proportions (wt.%) used in the mixture (M) were 1/5 Guadix clay and 4/5 La Gabia clay, following the suggestions of current manufacturers. Four types of bricks were prepared:

(a) bricks made up with clay mixture (M) plus 10 wt.% calcite in grains with particle size $0.5 \leqslant Ø_G \leqslant 2$ mm, to reproduce the composition of ancient bricks previously studied [13], denotes as MGW;
(b) bricks made up with clay mixture (M) plus 10 wt.% calcite powder (P) with particle size $Ø_P \leqslant 0.1$ mm, denotes as MPW;
(c) similar as in (a) but with 0.5 wt.% sodium chloride (S), denoted MGS, according to some authors [14,15], who demonstrated that this salt with this percentage would prevent the lime blowing of bricks containing up to 10% calcite in grains;
(d) similar as in (b) but with 0.5 wt.% sodium chloride (S), denoted MPS.

Samples were fired at different temperatures (850, 1000 and 1100°C) in a muffle furnace (Herotec CR-35, Spain).

The grain size of the two raw materials was determined using a computerised laser instrument (Galai CIS-1, Israel).

The mineral phases of the raw materials as well as the mineralogical changes taking place upon firing were identified by a diffractometer (Philips PW 1710, Holland) with graphite monochromater. Unoriented crystalline powder was used. Samples were ground in an agate mortar and separated to <50 μm. Analysis conditions were: 0,1 2θ/s goniometer speed, CuK_α radiation, 40 kV, 40 mA and 3–60° (2θ) explored area. XRD analysis was carried out with the PLV program [16].

Ultrasound testing was performed using an apparatus with a pulse frequency of 100 kHz (Steinkamp BP-5, Germany). The transductors were 2.7 cm in size. The propagation velocity of ultrasonic pulses has been measured by direct transmission in accordance with ASTM D 2845 normative [17] on dry test samples. These data were used to obtain information on the degree of compactness of the bricks after their manufacture. The velocity of the ultrasound waves were determined under controlled thermohygrometric conditions ($T = 20$°C and HR = 50%). Calculated values were V_{P1}, V_{P2} and V_{P3}, which are the ultrasound propagation velocities measured in three orthogonal directions. V_{P1} corresponds to the shortest distance (between the faces and perpendicular to the "layering", the orientation of the clay sheets comprising the brick). V_{P2} is the medium distance (between the ends) and V_{P3} is the longest distance (between the sides), in accordance with Regulation UNE 67-019-93 [18].

The structural anisotropy of the bricks was calculated from the above velocities using Eq. (1) [19]:

$$\Delta M_{i,jk} = 100 \times \left(1 - \frac{2V_{Pi}}{V_{Pj} + V_{Pk}}\right) \quad (1)$$

The stress tests were performed with a press with a 300 T capacity (Metro Com MI 30, Italy). The samples (3×3×3 cm) were subjected to a dry uniaxial unconfined stress, exerting pressure perpendicular to the layering of the brick, according to ASTM D 2938 regulation [20]. This is expressed as the relationship between the maximum force of the sample (F) and the surface on which the force is applied (A_{Pi}). Thus, the resistance of the brick is defined by Eq. (2):

$$R_{Pi} = \frac{F}{A_{Pi}} \quad (2)$$

We propose combining the results of the two above measuring techniques to define a new quantitative parameter termed the resistance anisotropy, R_{Ai}. This parameter, as expressed in Eq. (3),

$$R_{\mathrm{Ai}} = \frac{R_{\mathrm{Pi}}}{\Delta M_{\mathrm{i,jk}}} \tag{3}$$

is the ratio between the resistance of the brick and its structural anisotropy, defined respectively by Eqs. (2) and (1). It has been established to simplify the interpretation of the results. Thus, the technical quality of a brick is thus related to high values of resistance anisotropy.

In this paper we study the resistance anisotropy in the direction perpendicular to the layering since the clay minerals comprising the greater part of these bricks have a marked lamination, increased during the moulding and drying, producing notable anisotropy. We have, therefore, used the following resistance anisotropy equation:

$$R_{\mathrm{A1}} = \frac{R_{\mathrm{P1}}}{\Delta M_{1,23}} \tag{4}$$

Thus defined, its use as a parameter to measure the quality of a brick at the end of the manufacturing process is given by the relation of the physical stress produced on the brick with a mathematical parameter (its anisotropic structure). Thus, the quality of a brick is conditioned by its structural anisotropy for a constant mechanical stress. It is not applicable for small mechanical stresses, since in that case, although the isotropy may be very high $\Delta M \cong 0$, the quality of the brick cannot be determined ($\Delta M \cong 0$ does not reveal whether the brick will break or not).

Variance analysis (ANOVA) has been used to interpret the data as it allows the determination of which of the factors and the possible interactions between them are significant [10]. A two-way ANOVA was therefore selected to determine the significance of the factors (clay type, addition/absence of additives, and temperature) and the two-factor-effect interactions. Results, corresponding to the resistance anisotropy of each brick, were treated using the statistical package Statistica [21].

3. Results and discussion

3.1. Mineralogical composition

The study of grain sizes from 1 to 20 μm (Fig. 1) shows that the mixture used in the manufacturing process results in a grain-size distribution basically between 1 and 7 μm.

XRD data show that the raw material from the mixture fundamentally consists of quartz and lesser amounts of calcite, feldspars and phyllosilicates (illite, paragonite, chlorite, kaolinite and smectite). The mineralogical composition of the bricks is given in Figs. 2 and 3. The minerals are identified as follows: quartz, the most abundant, potassium feldspar and hematites. There are also small amounts of illite, phyllosilicates, gehlenite, anorthite, wollastonite, diopside, halite and portlandite.

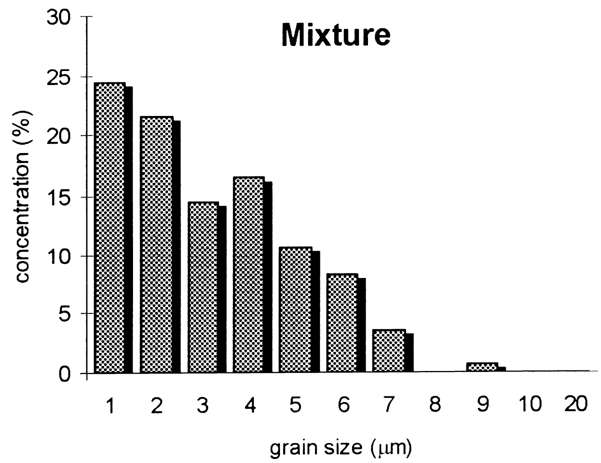

Fig. 1. Grain-size distributions for mixture sample. The horizontal bar represents an interval of 20 μm and the height shows the observed frequency.

The diffractograms reveal that, with increasing temperature, the phyllosilicate peaks, including illite, decrease in intensity until disappearing before 1100°C. At 1000°C wollastonite, diopside and gehlenite appear, typical high-temperature minerals [23,24]. The quartz content remains constant, while the presence of hematites increases. The feldspar peak also increases and at 1100°C shifts to a more anorthitic composition, partially due to gehlenite decomposition [25].

Samples with added sodium chloride, MPS and MGS, show a halite peak, while those containing coarse calcite, MGW and MGS, reveal portlandite peaks due to the transformation of $CaCO_3$ into CaO, who readily reacts with humidity and transforms into $Ca(OH)_2$ during cooling [26].

3.2. ANOVA study

Working with the samples obtained using grain particle sizes was impossible to obtain measurements neither structural anisotropy nor stress tests, due mainly to: (a) the reduction in the plastic contraction, that do not permit to do stress test, and (b) the increase in the porosity of the brick that do not permit to obtain real values of structural anisotropy.

The data used in the ANOVA are those obtained only with the powder particle sizes which are summarized in Tables 1 and 2.

The statistical study reveals that neither sodium chloride content nor temperature are significant because

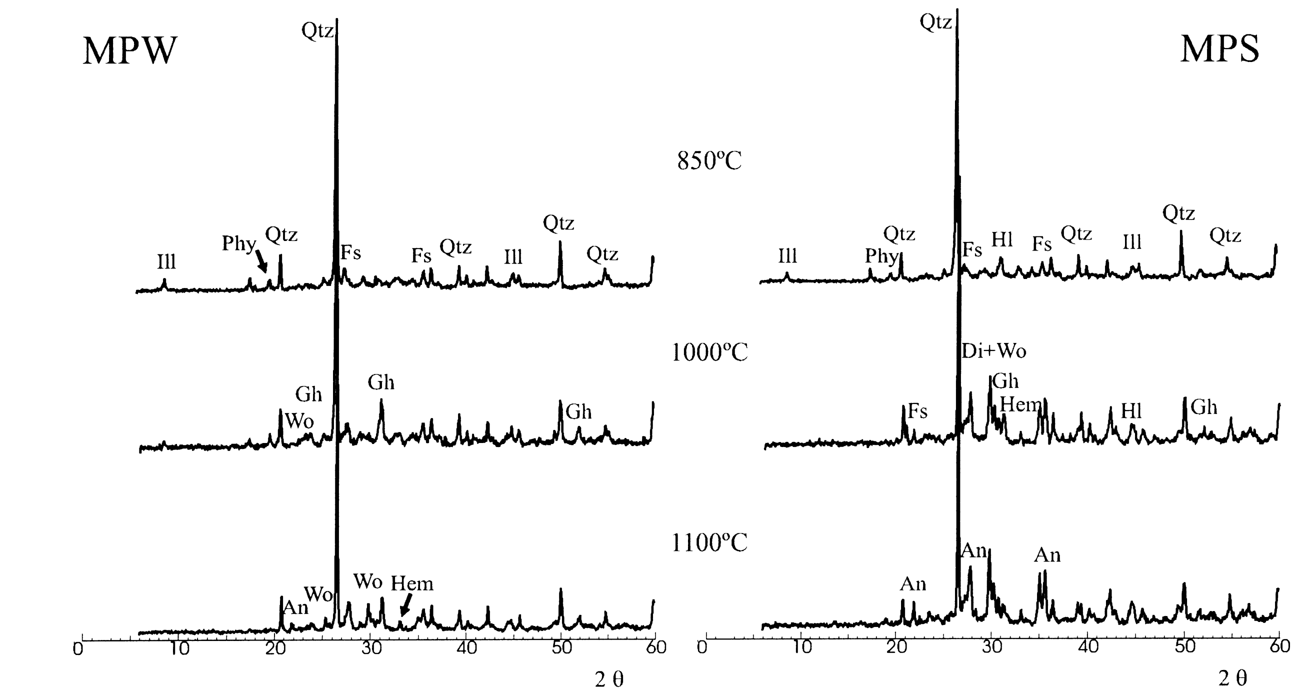

Fig. 2. Powder calcite bricks X-ray diffraction patterns, without sodium chloride (MPW) and with sodium chloride (MPS). Legend (mineral symbols after Krez [22]): Qtz = quartz, Phy = fillosilicates, Ill = illite, Fs = feldspars, Hl = halite, Wo = wollatonite, Gh = gehlenite, Hem = hematite, An = anortite).

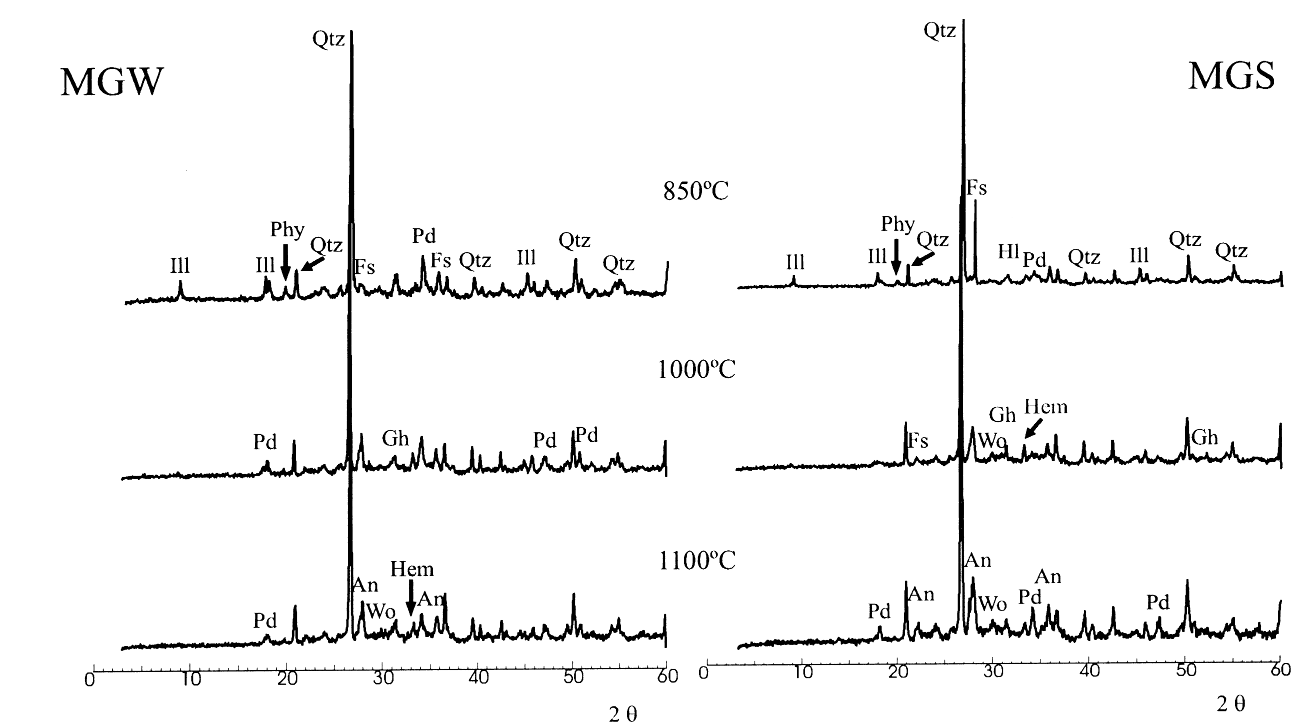

Fig. 3. Grain calcite bricks X-ray diffraction patterns, without sodium chloride (MGW) and with sodium chloride (MGS). Legend (mineral symbols after Krez [22]): Qtz = quartz, Phy = fillosilicates, Ill = illite, Fs = feldspars, Ha = halite, Pd = portlandite, Wo = wollatonite, Gh = gehlenite, Di = diopside, Hem = hematite, An = anortite).

Table 1
Dry uniaxial unconfined stress exerting and propagation velocity of ultrasonic waves to the three directions of the bricks

	R_{P1} (kg/cm^2)	R_{P2} (kg/cm^2)	R_{P3} (kg/cm^2)	V_{P1} (m/s)	V_{P2} (m/s)	V_{P3} (m/s)
MPW (850°C)	93.09	130.14	126.52	1440	2103	1912
MPW (1000°C)	129.94	157.50	154.26	1732	2338	2131
MPW (1100°C)	112.69	239.16	243.46	2147	2934	2662
MPS (850°C)	81.49	70.97	69.14	1479	2170	2160
MPS (1000°C)	84.09	100.12	91.60	1757	2315	2166
MPS (1100°C)	83.99	142.00	137.45	1880	2857	2725

Table 2
Values of R_{P1}, $\Delta M_{1,23}$ and R_{A1}[a]

	R_{P1} (kg/cm^2)	$\Delta M_{1,23}$ (%)	R_{A1} (kg/%cm^2)
MPW (850°C)	93.09	28.27	3.29
MPW (1000°C)	129.94	22.49	5.78
MPW (1100°C)	112.69	23.27	4.84
MPS (850°C)	81.49	31.69	2.57
MPS (1000°C)	84.09	21.58	3.90
MPS (1100°C)	83.99	32.64	2.57

[a] Data used in ANOVA analysis.

Table 3
Multiple analysis (method of LSD, 95%)

Factor	Level	Count	LS mean	P-value (%)	Group[a]	
					Salt content	Temperature
Salt Content[b]	W	3	4.637	7.33	X	
	S	3	3.013		X	
T (°C)	850	2	2.930	14.97		X
	1000	2	4.840			X
	1100	2	3.705			X

[a] Off-set columns denote a statistically significant difference.
[b] W, without sodium chloride; S, with sodium chloride.

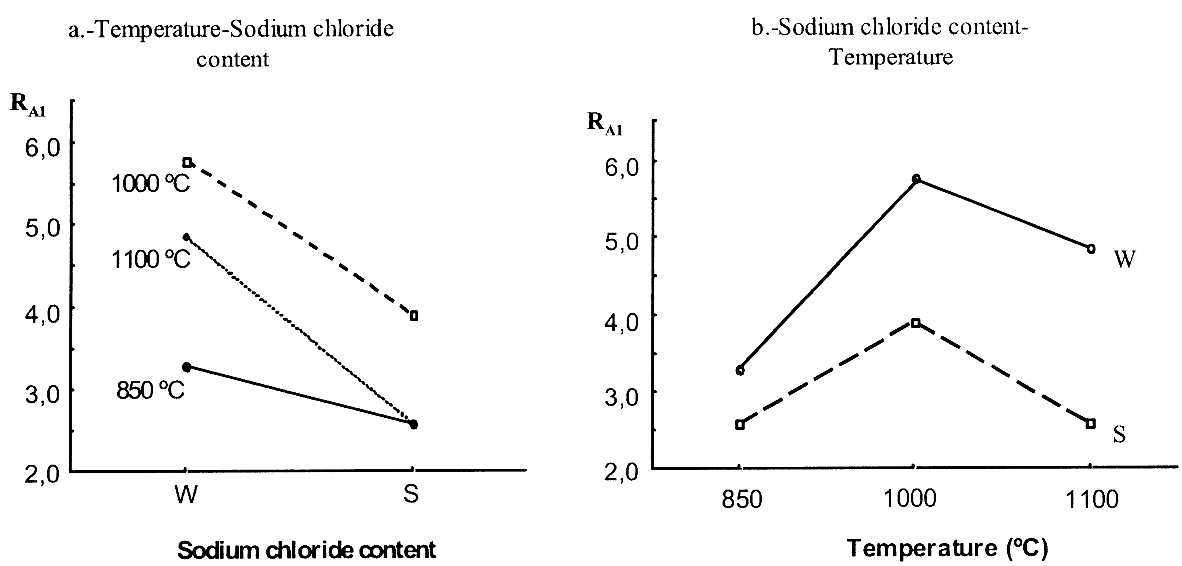

Fig. 4. The two-factor interactions: (a) temperature; (b) sodium chloride.

they give a P-value $>5\%$ (Table 3). This means that the resistance anisotropy does not depend on the values of the firing temperature and the addition/absence of additives in the interval tested.

Graphs showing the two factor interactions are given in Fig. 4. From Fig. 4a and b it can be observed that the best results of resistance anisotropy are obtained for the temperature of 1000°C and when no NaCl is added to the mixture.

4. Conclusions

As is well known, the quality of a brick is basically determined by the type of clay (raw material) used in its manufacture. As revealed by this study, the use of a parameter as anisotropy resistance permits the evaluation of the brick quality due to the summarizing of the effects of mechanical resistance and structural anisotropy obtained when a powdered calcite form is used as the raw material, so a great value of this variable indicates less anisotropy and greater mechanical resistance (a good compaction).

On the other hand, the same variable can be used to evaluate the effects of firing temperature and the absence/addition of sodium chloride. Although these variables seem to have no significant influence, high values of resistance anisotropy are obtained in bricks without sodium chloride and at 1000°C. In short, the resistance anisotropy of the bricks is, in increasing order: MPS and MPW. These data are in complete agreement with those obtained with other techniques (e.g. hydric tests on absorption, desorption and capillarity, porosimetry, accelerated ageing tests in the laboratory, etc.) [27], and confirming the resistance anisotropy as a feasible quantitative parameter for use in controlling the quality of the manufacturing process of bricks.

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