Estimated and real durability of unfired clay bricks: Determining factors and representativeness of the laboratory tests

A. Seco, P. Urmeneta, E. Prieto, S. Marcelino, B. García, L. Miqueleiz

Abstract

This paper presents an analysis of the representativeness of the main laboratory tests and the real durability of earth-based construction materials. For this study, a natural marl soil, mixed with different percentages of silica sand, was treated with portland cement, hydraulic lime, a mix of lime and ground granulated blastfurnace slag and other binder composed of a high magnesium oxide waste mixed with ground granulated blastfurnace slag. All the combinations were characterized based on the usual durability related laboratory tests as are: maximum density, unconfined compressive strength, wetting and drying, Swinburne accelerated erosion resistance, capillarity water absorption, total water absorption and freeze/thawing cycles. The results of these tests have been related to the real durability of the samples for eighteen months of outdoor exposure. They revealed the positive effect of sand adding in the materials durability and the great result of the binder based on magnesium oxide with ground granulated blastfurnace slag. It was also demonstrated the representativeness of the water absorption test as a durability indicator of earth based construction materials durability.
cementitious additives, is an effective and economical way to improve the material engineering properties, due to the cementitious gels formation which develop a resistant matrix around the soil particles. This increases the treated soil density, improving contact between particles, thus increasing its mechanical properties [12,15]. In addition the cementitious additives used, improves the clay soils workability by decreasing their plasticity [19,7] and decreases the water movement capacity, improving its durability [2,9,13]. The use of a high energy compaction in manufacturing improves the contact between particles, increases the material density and reduces the size and number of pores. This improves the particle mechanical properties and contributes to make them less susceptible to the water effects [4].

Related to the granulometry modification convenience, an experts agreement hasn’t been reached: many authors consider that the sand addition to soils with which these materials are made, allows to improve their properties since it increases the product density and decreases the amount and size of the mix pores, which could be an indirect indicator of good mechanical properties and durability. In this sense, different manuals and published studies recommend the use of different proportions sand to clay. For example, Muntohar [12] stated the optimum proportion soil to sand was 70 to 30% to get the higher compressive strength, higher density and lower permeability in a stabilized soil. Ciancio and Walker [3] tested ten soil mixes for the stabilized with cement and not stabilized rammed earth construction. In these mixes the clay and sand contents were 5–40% and 20–60% respectively. They found that six of the mixes were adequate for this purpose based on unconfined compressive strength, accelerated and drying shrinkage tests. The four mixes were not suitable were those with the highest clay content. On the other hand, some authors working with pure clay soils achieved materials with high mechanical properties and durability. Oti et al. [14] and Oti and Kinuthia [17] stabilized pure Lower Oxford Clay soils with Portland cement and lime plus Ground Granulated Blastfurnace Slag (GGBS). They obtained unconfined compressive strength values above 5 MPa and a highest weight loss at the end of 100 freezing-thawing cycles of 1.9%. This shows the complexity of the interpretation of the effect of the sand to clay content in the stabilization of a soil, and the difficulty of results extrapolation from one study to another. Anyway, it has been stated that sand addition allows a better soil workability and the swelling and shrinkage reduction when very plastic soils are used [3]. Finally, in EBCMs manufacturing it is important to consider the possibility to use products such as plasters or water repellents that avoid, together with the proper selection of the type and amount of cementitious additive, compaction energy and the soil particle size, the water to enter in the material.

Although there is no consensus in this regard, the relationship density-resistance-durability is often considered for the composition design and the selection of the EBCMs manufacturing parameters. This is why the technical characterization of these materials is based on tests of mechanical strength and water related properties, from which their durability is estimated. This way of durability estimation, commonly used in conventional building materials, has shown to be unreliable in the EBCM case, underestimating it many times, when compared with these materials real durability [6].

This work, aimed at an audience both scientific and technical, presents an experimental study which analyzes systematically the effect of main EBCMs manufacturing parameters affecting the durability, comparing the laboratory results with those which have been obtained under outside use conditions to establish the reliability of different laboratory tests to predict the real durability of an EBCM.

2. Materials

2.1. Soil

The soil used in this study was a sample of grey marl from the region of Pamplona, Northern Spain. This is a soft gray-colored rock, without defined stratification. Marl is a low load-bearing capacity soil, which greatly limits its use as construction material. In order to carry out this experiment, one tonne of natural marl was extracted, and after homogenization of the sample, it was crushed to a maximum particle size of 2 mm. The sample was characterized by determining its Atterberg limits following standards UNE 7378 and 103103, obtaining LL = 25.72% and PL = 18.05. Based on Casagrande Classification, this soil belongs to class CL, low-plasticity clayey silts. Chemical XRD analysis of this sample gave a mineralogical composition of 51% calcite, 20% illite, 15% quartz, 5% kaolinite, 5% attapulgite and 4% ankerite.

2.2. Sand

In this study a commercial silicon sand for mortars was used. This sand was obtained from the crushing of siliceous natural rocks. Fig. 1 shows the its grading curve.

2.3. Portland cement (PC)

Portland cement used in this study was manufactured in accordance with the European Standard EN 197–1, marketed under the trade name of CEM II B-M VL 52.5 N. Table 1 shows its composition as well as the other additives considered in this experiment, expressed as their most important oxides, based on XRF analysis.

2.4. Limes

Two different types of lime were used in this study: A Natural Hydraulic Lime (NHL-5), obtained from burned non-pure limestone and manufactured in accordance with the European Standard EN 459–1. This Lime has hydraulic properties due to the presence of Aluminum and Silicon oxides as well as Calcium oxide. Also a calcareous hydrated lime (CL-90-S) was used in the study, obtained from burned pure limestone and manufactured in accordance with the European Standard EN 459–1.

2.5. PC-8

This material is a byproduct rich in Mg, obtained during the calcined magnesite production by means of the calcination of natural MgCO3 rocks up to 1100 °C. This process is carried out in a rotatory...
kiln with crosscurrent air circulation, which pulls dust particles along the whole kiln. So that, this dust contains MgCO₃ (inert), calcined MgO (reactive) and vitrified MgO (inert) particles.

2.6. GGBS

GGBS is a by-product obtained during the manufacturing of pig iron. It has a big cementitious potential because of its richness in calcium, silicon and aluminum oxides. It was used combined with the calcareous hydrated lime and the PC-8 as activators with a ratio of 20% CL-90-S or PC-8/80%GGBS.

3. Methods

3.1. Samples preparation

For each soil and sand combinations, Optimum Moisture Content (OMC) corresponding to the compaction energy, was stated in accordance with the procedure defined in the Spanish Standard UNE 103500. After that, the mixings of soil, sand and additives were carried out in an industrial mixer until their complete homogenization and OMC was added slowly to get its uniform distribution. After a wet mixing time, the mixes were treated in a high speed homogenizer to guarantee the goodness of mixing and moisture distribution. Once the quality of the mixes was visually verified, 65 mm diameter and 75 mm height cylindrical samples were prepared, pressing the material in a mold at 8 MPa. The samples were immediately unmolded after fabrication and the curing time, since the fabrication till the 28 days testing age, was carried out in a wet chamber at 20°C and 100% HR.

All mix combinations contained a 10% binder and 90% target material, in which variable sand percentages were included. Table 2 shows the different chosen mixes, based on the bibliography.

Table 2
Tested combinations.

<table>
<thead>
<tr>
<th>Combination</th>
<th>Sand (%)</th>
<th>Binder</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>Portland Cement</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>Portland Cement</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>Portland Cement</td>
</tr>
<tr>
<td>4</td>
<td>50</td>
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</tr>
<tr>
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<td>0</td>
<td>Hydraulic lime</td>
</tr>
<tr>
<td>6</td>
<td>10</td>
<td>Hydraulic lime</td>
</tr>
<tr>
<td>7</td>
<td>30</td>
<td>Hydraulic lime</td>
</tr>
<tr>
<td>8</td>
<td>50</td>
<td>Hydraulic lime</td>
</tr>
<tr>
<td>9</td>
<td>0</td>
<td>CL-90-S+GGBS</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>CL-90-S+GGBS</td>
</tr>
<tr>
<td>11</td>
<td>30</td>
<td>CL-90-S+GGBS</td>
</tr>
<tr>
<td>12</td>
<td>50</td>
<td>CL-90-S+GGBS</td>
</tr>
<tr>
<td>13</td>
<td>0</td>
<td>PC-8+GGBS</td>
</tr>
<tr>
<td>14</td>
<td>10</td>
<td>PC-8+GGBS</td>
</tr>
<tr>
<td>15</td>
<td>30</td>
<td>PC-8+GGBS</td>
</tr>
<tr>
<td>16</td>
<td>50</td>
<td>PC-8+GGBS</td>
</tr>
</tbody>
</table>

3.2. Tests

Tests were carried out in accordance with the Spanish Standard UNE 41410 for the unfired clay bricks characterization at the age of 28 days. To ensure the representativeness of each of them, 4 specimens were tested each time. The maximum density was obtained, together with the OMC, based on the Spanish Standard UNE 103500. The mechanical properties characterization of the samples was carried out according to the unconfined compressive test defined in the Spanish Standard UNE 103400. The capillary water absorption and the total water absorption tests were carried out based on the European Standard UNE EN 772–11. The freeze/thawing cycles resistance was established based on the Spanish Standard UNE 67028 EX and it was realized with samples both normal and surface treated with a commercial water repellents for façades. Besides, samples of all the combinations, with and without water repellent treatment, were placed outdoors and exposed to weather conditions making a monthly visual inspection of the observed damages in specimens during 18 months.

4. Results and discussion

4.1. Maximum density and OMC

Fig. 2 shows the curves density–water content for each soil-sand mixes.

It can be seen that for each combination a characteristic curve was obtained, which determines the optimum water content to maximize the efficiency of the compaction energy. As sand content increased maximum density increased and OMC decreased from 2.04 g/cm³ and 11.6% in the case of the soil alone until 2.11 g/cm³ and 8.6% of the mixture with 50% of sand, respectively.

4.2. Unconfined compressive strength

Fig. 3 shows the unconfined compressive strength obtained values for every combination at the age of 28 days.

The differences in resistance obtained depended mainly on the binder type and not on the sand percentage as it can be seen. This fact contradicts the existence of a relationship density-resistance in this soil. Additives that showed better results were PC and the PC-8+GGBS, with values between the 11.1 and the 13.7 MPa, with slight oscillations with different sand percentages. CL-90-S+GGBS showed slightly lower values, between 10.2 and 11.7 MPa. Finally NHL-5 got values between 4.4 and 5.5 MPa which, although they were values much lower than the other additives, also overcome...
1.3 MPa, lowest reference value allowed in the Spanish Standard UNE 41410 for the EBCMs.

4.3. Wetting-drying cycles and Swinburne accelerated erosion

After the wetting–drying cycles test, none of the tested specimens corresponding to the different combinations showed cracks, swelling, losses of layers or any other surface defect, keeping intact after 6 cycles of wetting and drying that establishes the Spanish Standard UNE 41410, therefore all combinations passed this test.

In the case of the accelerated erosion Swinburne test, no one of the samples presented neither holes nor any other kind of damages after the 10 min test duration, therefore all combinations were considered suitable also for this test.

4.4. Capillarity water absorption

As it could be seen in Fig. 4, there are big differences regarding to the capillarity water absorption for each one of the additives along the 10 min test. The sand percentage of the mix also showed a clear effect on these results.

In every case, the water absorption values increased as the sand percentage did. The additive that absorbed more water was the NHL-5, with a maximum absorption of 1.9%, followed by the CL-90-S+GGBS and the cement with 1.8% and 1.3%, respectively, all of them for the 50% sand. In this test, the binder with the best behavior was the PC-8+GGBS, showing not only the lowest values of capillarity absorption but also the lower increase for all the different contents of sand, with a 0.7% for the soil alone and only a 0.8% absorption with a 50% sand.

4.5. Total water absorption

This test stated the water absorption by the materials after a 24 h water immersion. Fig. 5 shows the results obtained for each tested combinations.

As it can be seen, and in contrast with what was observed in the capillarity absorption test, in this case the absorption values were much higher, probably due to the much greater test duration. Another significant difference was how in this test water absorption values decreased as the percentage of sand increased. This could be due to the test duration which allowed the samples saturation. Additionally these tests demonstrated that as the sand percentage in samples increased, capillary water absorption was faster but as the sand has a lower affinity for the water than clay, the total amount of absorbed water was smaller. As in the capillarity test, the additive which behaves worse was NHL-5, followed by PC and the CL-90-S+GGBS, being PC-8+GGBS the binder with the best performance.

4.6. Freezing/thawing cycles test

In this test, the appearance of the samples after 25 cycles of freezing and thawing was analyzed. Samples with and without water-repellent treatment were tested, and the appearance of damages during the test time was analyzed. After 12 cycles, a first observation was carried out and damages were detected only in the combinations without waterproofing treatment of NHL-5 with 0, 10, and 30% of sand.

After the 25 cycles, damages were observed in all the specimens of NHL-5 without water-repellent treatment, as well as in the NHL-5 samples with water-repellent treatment with 0, 10, and 30% sand. In the rest of the samples, cracks or other surface damages were not detected. These results highlight the efficiency of the other binders regarding to the NHL-5 and the low ability of this test to state the effectiveness of the surface water-repellent treatment.

4.7. Outdoors exposition

In contrast to the laboratory tests, samples of all combinations were kept outdoors during 72 weeks between December 1, 2014 and May 31, 2016 to state the effect of the local meteorology in the real durability of the different combinations. Fig. 6 shows the meteorologic conditions during the outdoor exposition period. At this period there was a total rainfall of 1202 mm, temperatures exceeded 30 °C 43 days and 60 days were reached temperatures below 0 °C.

During the test time the observed damages mainly consisted of firstly radial cracks in the upper contour of the samples, followed...
by the loss of material, probably helped by the water accumulation in this face during the rain events. These damages increased over time until the sample has a “bullet” shape, as it can be seen in Fig. 7 although the damaged sample criterion was the appearance of surface cracks.

Dates of failure of different combinations as well as those that resisted the 18 months without loss of material or other surface failures are shown in Tables 3 and 4.

Damages in the samples without waterproofing of NHL-5 with 10% sand were observed on February 2015 (week 9). On April 2015 (week 17), there were observed damages in the samples without waterproofing of cement with 0% sand and in PC water proof treated samples with 0 and 10% sand.

On May 2015 (week 21) damages appeared in the samples without waterproofing of NHL-5 with 0% sand, PC without water-repellent with 10% sand and CL-90+GGBS without water-repellent treatment with 0% sand. On June 2015 (week 25) only were detected damages in the samples of NHL-5 with water-repellent treatment without sand.

On July 2015 (week 29), significant damages were observed in NHL-5 specimens without water-repellent with 30 and 50% sand and the water-repellent treated NHL-5 samples with 10–30% sand. On August 2015 (week 33), surface damages in water-repellent treated NHL-5 specimens with 50% sand were detected.

The following surface damages were detected in October 2015 (week 41) in CL-90-S+GGBS combinations without water-repellent with 10, and 30% of sand, in the CL-90-S+GGBS with water repellent treatment without sand, as well as in samples of PC without water-repellent treatment with 30 and 50% sand and in the water repellent treated ones with 30% sand.

On November 2015 (week 45), water repellent treated PC with 50% sand samples failed. On December 2015 (week 49), cracks in

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**Fig. 6.** Meteorologic conditions during the outdoor exposure period.

**Table 3**

<table>
<thead>
<tr>
<th>Without water repellent treatment</th>
<th>0% Sand</th>
<th>10% Sand</th>
<th>30% Sand</th>
<th>50% Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland cement</td>
<td>04/15</td>
<td>05/15</td>
<td>02/16</td>
<td>04/16</td>
</tr>
<tr>
<td>NHL-5</td>
<td>05/15</td>
<td>02/15</td>
<td>07/15</td>
<td>07/15</td>
</tr>
<tr>
<td>CL-90+GGBS</td>
<td>05/15</td>
<td>10/15</td>
<td>10/15</td>
<td>10/15</td>
</tr>
<tr>
<td>PC-8+GGBS</td>
<td>R</td>
<td>R</td>
<td>R</td>
<td>R</td>
</tr>
</tbody>
</table>

**Table 4**

<table>
<thead>
<tr>
<th>With water repellent treatment</th>
<th>0% Sand</th>
<th>10% Sand</th>
<th>30% Sand</th>
<th>50% Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland cement</td>
<td>04/15</td>
<td>04/15</td>
<td>03/16</td>
<td>04/16</td>
</tr>
<tr>
<td>NHL-5</td>
<td>06/15</td>
<td>07/15</td>
<td>07/15</td>
<td>08/15</td>
</tr>
<tr>
<td>CL-90+GGBS</td>
<td>10/15</td>
<td>03/16</td>
<td>R</td>
<td>R</td>
</tr>
<tr>
<td>PC-8+GGBS</td>
<td>R</td>
<td>R</td>
<td>R</td>
<td>R</td>
</tr>
</tbody>
</table>

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**Fig. 7.** Appearance of samples during the outdoor exposition test. a) undamaged sample, b) damaged sample and c) sample with bid damages “bullet” shape.
the CL-90-S+GGBS specimens without treatment with 50% of sand were detected. Last samples failures, corresponding to the combination of CL-90-S+GGBS with water repellent treatment with 10% of sand were detected on January 2016 (week 53). Combinations that resisted the whole outdoors time were all with PC-8+GGBS as additive, water repellent treated and untreated, for all sand contents, as well as the CL-90-S+GGBS combinations with water repellent treatment and 30–50% sand.

If we analyze the general behavior of the samples over time, it could be seen that from the total of 32 combinations tested, in the first 24 weeks of outdoor exposure, they failed a total of 7 combinations, of which 4 are combinations of NHL-5 without the water-repellent, clearly indicating a worse behavior of this additive in relation to the outdoor exposure.

Between 24 and 48 weeks of exposure defects in a total of 12 combinations have been generalized: Although there is not a very clear pattern, it can be observed that among these combinations additives, there is a decreasing trend for the resistance between CL-90-S+GGBS, PC and NHL-5, as well as a slight positive effect of the sand content of the sample and of the water repellent treatment.

Between 52 and 72 weeks, only the failure of two combinations of CL-90-S+GGBS, in the weeks 52 and 56, were detected. From this moment until the 72th week, there were no more failures, resisting the test all combinations of PC-8+GGBS and two CL-90-S+GGBS.

5. Conclusions

This experimental study allowed the following conclusions:

1. The sand content of samples does not have a relationship with their mechanical resistance, which clearly depends on the type of the used additive.
2. Although all combinations widely exceeded the minimum value of unconfined compressive strength established in the Spanish Standard, in this test, the best results were obtained with the PC-8+GGBS and PC, followed closely by the CL-90-S+GGBS and lesser by the NHL-5. If the resistance of each additive is compared with the outdoor observed durability, it can be stated that the relationship resistance–durability was met on all additives except for PC, which overestimates it.
3. Wetting and drying cycles and Swinburne accelerated erosion tests demonstrated to be low demanding test and to have no agreement with the observed durability so, they can not be considered adequate test for the EBCMs durability estimation.
4. In the capillarity absorption test, the result of the behavior of additives fits to the pattern of the outdoor observed durability: lowest water absorptions were obtained with the PC-8+GGBS, followed by the PC and the CL-90-S+GGBS and finally the NHL-5. However, the increase in the samples sand content increased significantly the capillarity absorbed water, which provides an opposite result to the observed durability, which increased with the percentage of sand. This indicates that this test is not appropriated to estimate EBCMs real durability.
5. In the case of the total water absorbed test, both the additives behavior and the sand percentages, agree with the pattern observed in the outdoor test. This allows to establish the suitability of this test as a EBCMs durability estimator.
6. During the freeze/thawing cycles test, damages only happened in samples treated with NHL-5, first without water-repellent and then with the treatment, with a clear sand content influence in the resistance to the test. The fact that, any samples with other binders were not damaged, highlights the unsuitability of this test, or at least the number of cycles considered as representative by the Spanish Standard.
7. During outdoor exposure test, since the 56th week, no more samples were damaged. This shows that combinations that reached this age without surface damages have some properties that allow to suppose they are durable in outdoor conditions.

As a final conclusion of this experiment it could be emphasized the positive effect on materials durability of the sand addition, the good result of the binder consisting of PC-8+GGBS, as well as the representativeness of water absorption test as an EBCMs real durability estimator.

References
