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OPTIMIZATION OF BRICKS PRODUCTION BY EARTH HYPERCOMPACTION PRIOR TO FIRING

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28 **HIGHLIGHTS**

- 29 • Proctor compacted, hypercompacted and extruded earth bricks were manufactured.
- 30 • Earth bricks were fired at five temperatures: 280, 455, 640, 825 and 1000 °C.
- 31 • Thermal treatment was quick to save energy and time.
- 32 • Compressive strength, water durability and moisture buffering were investigated.
- 33 • Excellent properties were achieved for hypercompacted bricks with low firing times and
- 34 temperatures.

35 **ABSTRACT**

36 This paper presents an innovative method for the production of masonry bricks, which combines
37 earth compaction and quick firing at low temperatures. Earth bricks were manufactured according
38 to three different methods, i.e. extrusion, standard Proctor compaction and hypercompaction to 100
39 MPa. All bricks were fired inside an electrical furnace by rising the temperature at a quick rate of
40 about 9 °C per minute to 280, 455, 640, 825 and 1000 °C, after which the furnace was turned off
41 and left to cool to the atmosphere with the brick inside it. These firing temperatures and times are
42 significantly lower than those employed for the manufacture of commercial bricks, which are
43 typically exposed to a maximum of 1100 °C for at least 10 hours (Brick Industry Association,
44 2006). A testing campaign was performed to investigate the effect of quick firing on the porosity,
45 strength, water durability and moisture buffering capacity of the different bricks. Quick firing of
46 hypercompacted bricks at moderate temperatures, between 455 and 640 °C, is enough to attain very
47 high levels of compressive strength, between 29 and 34 MPa, with a good to excellent moisture
48 buffering capacity. These properties are better than those of commercially available bricks. The
49 strength of hypercompacted bricks further increases to 53 MPa, a value similar to that of high-
50 strength concrete, after quick firing at 825 °C. Earth densification prior to thermal treatment
51 therefore improves material performance while enabling a significant reduction of firing
52 temperatures and times compared to current bricks production methods.

53

54 **KEYWORDS**

55 Bricks production, firing treatment, pore size distribution, compressive strength, water durability,
56 moisture buffering capacity.

57

58 **INTRODUCTION**

59 Fired earth bricks are commonly employed for the construction of masonry structures despite their
60 relatively large energy and carbon footprints. Bricks exhibit large levels of embodied energy
61 because of their production method which consist in subjecting extruded earth blocks to very high
62 temperatures, up to 1100 °C, for a period between 10 and 40 hours (Brick Industry Association,
63 2006; Zhang, 2013; Murmu and Patel, 2018). This energy-intensive thermal treatment is necessary
64 to achieve adequate mechanical and durability characteristics for construction applications. Besides
65 high levels of embodied energy, bricks also exhibit a limited ability to absorb/release vapour
66 from/to the indoor environment, which reduces the hygro-thermal inertia of buildings walls and
67 encourages electrical air conditioning of dwellings (Morton et al., 2005; Rode et al., 2005). Finally,
68 upon demolition, fired bricks generate waste that is often disposed in landfills, thus resulting in
69 environmental pollution and loss of land (Bossink and Brouwers, 1996).

70 Most of the above limitations could be overcome by using raw (i.e. unfired) earth bricks, which are
71 manufactured with relatively little energy as shown by Little and Morton (2001) and Morel et al.
72 (2001). Raw earth also exhibits a strong tendency to adsorb vapour from humid environments and
73 to release it into dry environments while simultaneously liberating and storing latent heat thanks to
74 an open network of nanopores and the high specific surface of clay particles. This property
75 increases hygro-thermal inertia and helps smoothing daily fluctuations of humidity and temperature
76 inside buildings with a consequent improvement of occupant comfort and an associated reduction of
77 air conditioning needs (Houben and Guillaud, 1989; Allinson and Hall, 2010; Pacheco-Torgal and
78 Jalali, 2012; Soudani et al., 2016; Gallipoli et al., 2017; Soudani et al., 2017). Finally, raw earth is
79 an entirely natural material which can be easily recycled or safely disposed into the environment.

80 Despite the above advantages, raw earth is still regarded as an unviable material for mainstream
81 construction due to relatively low levels of water durability and strength. Recent research has

82 however shown that “hypercompaction” of earth to very high pressures (of the order of hundreds of
83 megapascals) can produce raw bricks with levels of strength and stiffness that are higher than those
84 of standard fired bricks (Bruno et al., 2017; Bruno et al., 2018). This is possible thanks to a
85 densification of the material down to a porosity of about 0.13, a value similar to that of shale rocks
86 (porosity is the ratio between pore volume and total volume). Unfortunately, this large increase in
87 strength and stiffness does not correspond to a similar gain of durability, especially when raw earth
88 comes into contact with liquid water. For this reason, chemical stabilizers such as cement or lime
89 are often added to the earth to improve mechanical characteristics (Walker and Stace, 1997; Bahar
90 et al., 2004; Guettala et al., 2006; Jayasinghe and Kamaladasa, 2007; Kariyawasam and Jayasinghe,
91 2016; Khadka and Shakya, 2016; Venkatarama Reddy et al., 2016; Dao et al., 2018). Unfortunately,
92 the addition of chemical stabilisers reduces the moisture buffering capacity and hygro-thermal
93 inertia of the material (Liuzzi et al., 2013; McGregor et al., 2014; Arrigoni et al., 2017) while
94 largely increasing the carbon footprint (Worrell et al., 2001). Alternative stabilisation methods are
95 therefore necessary to improve water durability without increasing the environmental impact of raw
96 earth. In this respect, the application of moderate heat has been considered in a small number of
97 studies as a possible stabilisation method but never in association with a high compaction effort.
98 Mbumbia et al. (2000) investigated the hydro-mechanical behaviour of extruded lateritic earth
99 bricks fired at 350, 550, 750, 850 and 975 °C for 4 and 8 hours. They observed that both mechanical
100 and durability properties improve as temperature increases while firing time has only a marginal
101 effect. These findings were further confirmed by Karaman et al. (2006), who fired pressed earth
102 bricks at temperatures ranging from 700 °C to 1100 °C for different times from 2 to 8 hours. They
103 concluded that temperature plays a key role in changing the physical and mechanical properties of
104 the bricks while firing time has little effect.

105 The present work investigates, for the first time, a brick manufacturing method that relies on earth
106 hypercompaction to generate very high levels of material strength followed by quick firing at low

temperatures and times to attain good water durability. The increase of strength produced by earth hypercompaction prior to firing reduces the demands on thermal treatment, whose only purpose becomes the enhancement of water durability. This allows a very significant reduction of both firing temperatures and times respect to the values proposed by Mbumbia et al. (2000) and Karaman et al. (2006). Moreover, quick firing has the advantage of preserving a considerable part of the moisture buffering capacity of raw earth with a consequent gain of hygro-thermal inertia respect to standard fired bricks.

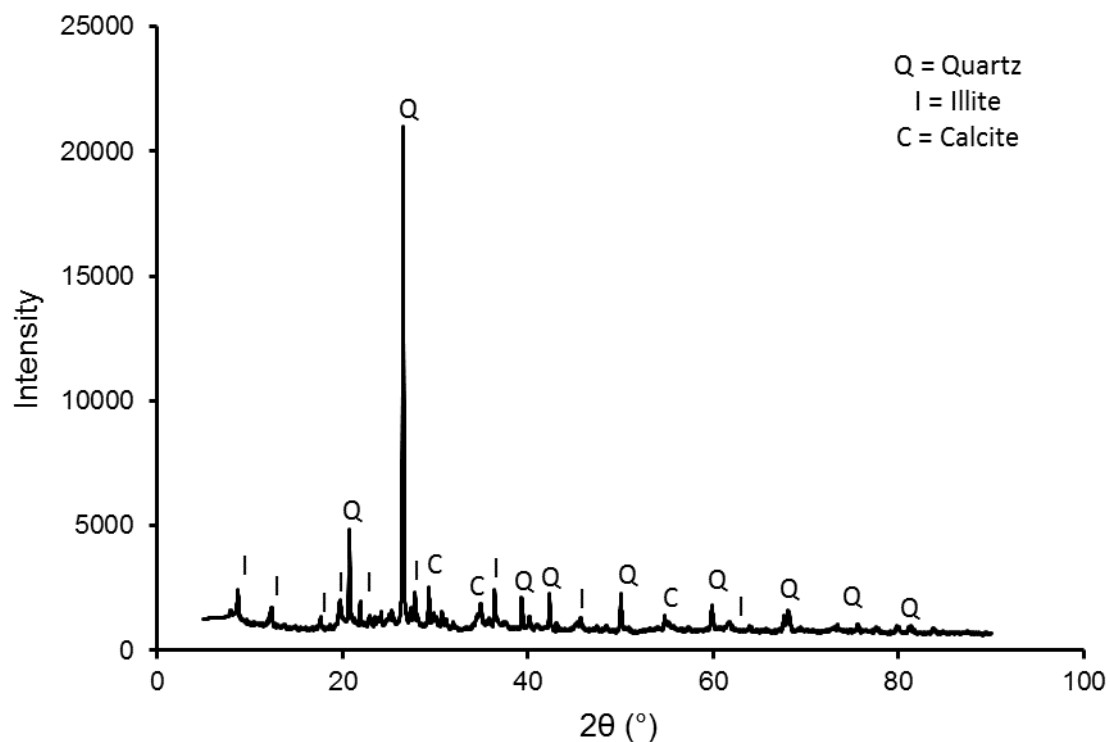
Quick firing is accomplished by placing a raw earth brick inside an electrical furnace and rapidly increasing the temperature to a given target, after which the furnace is switched off and allowed to cool to the atmosphere with the brick inside it. As shown later, a moderate temperature, between 455 °C and 640 °C, is already sufficient to ensure good levels of water durability. For hypercompacted bricks, this moderate temperature is also sufficient to generate a compressive strength of about 30 MPa, which is greater than the strength of most commercial bricks. Remarkably, if the hypercompacted bricks are quickly fired at a higher temperature of 825 °C, which is however still lower than the temperature imposed during current brick production, material strength increases to an extremely high value of 53 MPa.

The results obtained in the present work therefore indicate that a faster, cleaner and less energy-intensive thermo-mechanical process can be devised to improve production of masonry bricks while reducing environmental impact and increase efficiency. These preliminary results must however be supported by further investigation to quantify the ensuing energy savings and to extend the characterization of the hygro-mechanical and durability characteristics of the produced bricks.

MATERIAL AND METHODS

The earth used in the present work has been provided by the brickwork factory NAGEN from the region of Toulouse (South-West of France) and corresponds to a typical soil for the production of

131 standard fired bricks. The grain size distribution was determined by both wet sieving and
 132 sedimentation in compliance with the norms XP P94-041 (AFNOR, 1995) and NF P 94-057
 133 (AFNOR, 1992), respectively, which indicate that the material is composed by 40.8% sand, 42.9%
 134 silt and 16.3% clay. The Atterberg limits of the fine fraction (i.e. the soil fraction smaller than 400
 135 μm) were determined according to the norm NF P94-051 (AFNOR, 1993), which indicates a liquid
 136 limit of 33.0% and a plasticity index of 12.9%. These results classify the material as an inorganic
 137 clay of medium plasticity according to the Unified Soil Classification System USCS ASTM D2487-
 138 11 (2011). Both grain size distribution and plasticity properties also satisfy existing
 139 recommendations for compressed earth bricks (e.g. MOPT, 1992; Houben and Guillard, 1994;
 140 CRATerre-EAG, 1998; AFNOR, 2001) as discussed by Bruno (2016). Material mineralogy was
 141 investigated by means of X-ray diffractometry using an AXIS Nova X-Ray photoelectron
 142 spectroscopy (Kratos Analytica). Results from this test showed that the earth used in the present
 143 work is mainly composed of quartz, illite and calcite (Figure 1).



144
 145 *Figure 1. X-Ray spectrum of the base earth.*

146 Raw earth bricks were manufactured according to three different methods, namely extrusion,
147 standard Proctor compaction and hypercompaction. Both Proctor compacted and hypercompacted
148 bricks had dimensions of $200 \times 100 \times 50 \text{ mm}^3$, while extruded bricks had slightly larger dimensions
149 of $220 \times 110 \times 50 \text{ mm}^3$. This small variation was the consequence of the different sizes of the screw
150 press ejector of the extruded bricks and the compaction mould of Proctor and hypercompacted
151 bricks. A brief description of the three manufacturing processes is given below:

- 152 • Extrusion. Extruded bricks were manufactured by the brickwork factory NAGEN according
153 to the same process used for standard bricks. The dry earth was passed through a grinder and
154 sieved to remove grains larger than 1 mm. The sieved earth was subsequently mixed with an
155 optimum water content of about 18% and conveyed to a screw extruder with a rectangular
156 ejector section of $110 \times 50 \text{ mm}^2$. Finally, the extruded strip was cut into individual bricks
157 with length of 220 mm.
- 158 • Standard Proctor compaction. The dry earth was mixed at the optimum water content of
159 13.5%, which had been previously determined by standard Proctor compaction of samples at
160 different water contents (AFNOR, 1999). The moist earth was stored inside two plastic bags
161 for at least 24 hours to ensure the equalisation of pore water pressures. The equalised earth
162 was subsequently placed inside a stiff rectangular mould, with a horizontal cross section of
163 $200 \times 100 \text{ mm}^2$, and statically compacted to a target height of 50 mm by a piston with a
164 displacement rate of 0.1 mm/s. The amount of earth placed inside the mould was calculated
165 to attain a dry density of 1860 kg/m^3 , which corresponds to the Proctor optimum.
- 166 • Hypercompaction. The dry earth was mixed at the optimum water content of 5.2%, which
167 had been previously determined by static compaction to 100 MPa of samples at different
168 water contents (Bruno, 2016). The moist earth was stored inside two plastic bags for 24
169 hours to ensure equalisation before being compacted to 100 MPa with a rate of 0.17 MPa/s,
170 which resulted in a very dense material with an average porosity of 0.13. The earth was

“double compacted” by two pistons acting at the top and bottom of a “floating mould” with a horizontal cross section of 200 x 100 mm². The floating mould was supported by internal friction with the lateral surface of the brick. Double compaction is preferable to single compaction because it reduces frictional effects on the lateral brick surface and therefore increases the uniformity of stress and porosity inside the material. Double compaction could, however, only be employed for hypercompacted bricks because, for Proctor compacted bricks, the applied pressure was too low to generate enough lateral friction to support the weight of the floating mould. Further details about the hypercompaction procedure can be found in Bruno (2016).

After manufacturing, all bricks were equalised to the laboratory atmosphere, corresponding to a temperature of about 25 °C and a relative humidity of about 40%, for a minimum of one week and until a constant mass was attained. During this time, the water content of the bricks reduced significantly attaining a stable value of about 3%. After equalisation, a set of bricks was kept inside the laboratory while another set was prepared for the subsequent firing stage by drying for 24 hours at 105 °C followed by 12 hours at 200 °C. This additional drying was necessary to avoid that the material exploded when fired at higher temperatures due to the expansion of entrapped vapour.

Bricks were then fired inside an electrical furnace at five different temperatures of 280, 455, 640, 825 and 1000 °C. In all cases, the temperature was increased with an approximately constant rate of 9 °C per minute, which was the fastest rate allowed by the furnace. Once the target temperature was reached, the furnace was turned off and left to cool overnight with the brick inside it. Figure 2 shows the variation of temperature with time during both heating and cooling stages.

After firing, bricks were again equalised to the laboratory atmosphere (temperature of 25 °C and relative humidity of 40%) until a constant mass was recorded and, in any case, for not less than two weeks. Figure 3 shows both the dry density and the corresponding porosity (in bracket) of the bricks fired at different temperatures. The temperature of 25 °C refers to the unfired bricks, which were

196 simply equalised to the laboratory atmosphere without any thermal treatment. The dry density, and
197 hence the porosity of the material, were calculated from the mass, volume and water content of the
198 bricks measured after equalisation. In particular, water content was determined by drying at 105 °C
199 for 24 hours three small fragments of about 50 grams each taken at different heights of the failed
200 bricks after mechanical testing. This procedure relies on the assumption that only negligible
201 changes in water content occur during mechanical testing.

202 As expected, hypercompacted bricks exhibit a higher dry density than Proctor and extruded bricks
203 due to their large compaction pressure. Inspection of Figure 3 also indicates that, for all brick types,
204 dry density decreases as firing temperature grows, especially beyond 455 °C. This result is in
205 contradiction with previous studies (e.g. Karaman et al., 2006) where dry density increased
206 monotonically with growing firing temperatures, which is explained by the quick temperature ramp
207 imposed to bricks in the present work. Quick firing, combined with the high quartz content of the
208 base earth (Figure 1), promotes a rapid vitrification of the brick surface (Cultrone et al., 2004). This
209 impermeable skin then causes the formation of internal “sacks” of carbon dioxide and water vapour
210 with a consequent increase of porosity. Instead, in earlier studies by Karaman et al. (2006) and
211 Mbumbia et al. (2000), a very slow heating rate of only 1°C per minute was applied, which
212 prevented the rapid formation of a vitrified skin and therefore facilitated the evacuation of carbon
213 dioxide and water vapour from the brick core during firing. Note that carbon dioxide and water
214 vapour are typically generated by the burn off of carbonaceous organic matter and the
215 dihydroxylation of structured water at temperatures higher than 550 °C (Karaman, 2006; Baccour et
216 al., 2009).

217 Quickly fired bricks were then tested to measure compressive strength, water durability and
218 moisture buffering capacity. Mercury intrusion porosimetry tests were also undertaken to analyse
219 the influence of quick firing on material fabric.

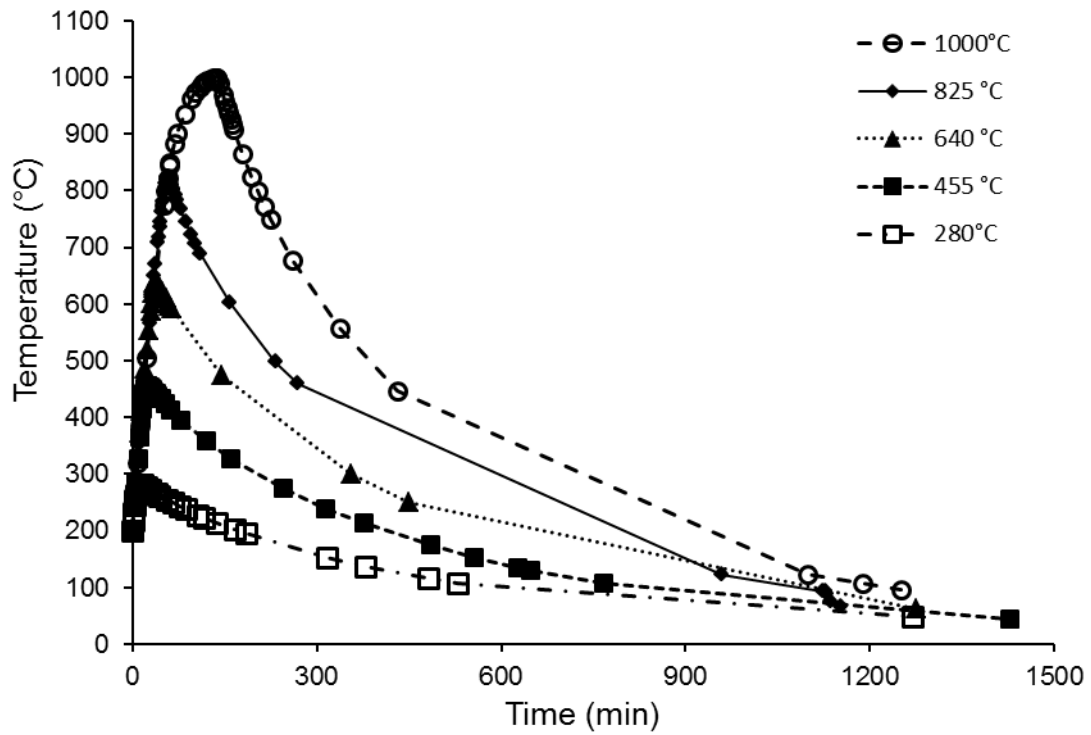


Figure 2. Quick thermal treatment: variation of firing temperature with time.

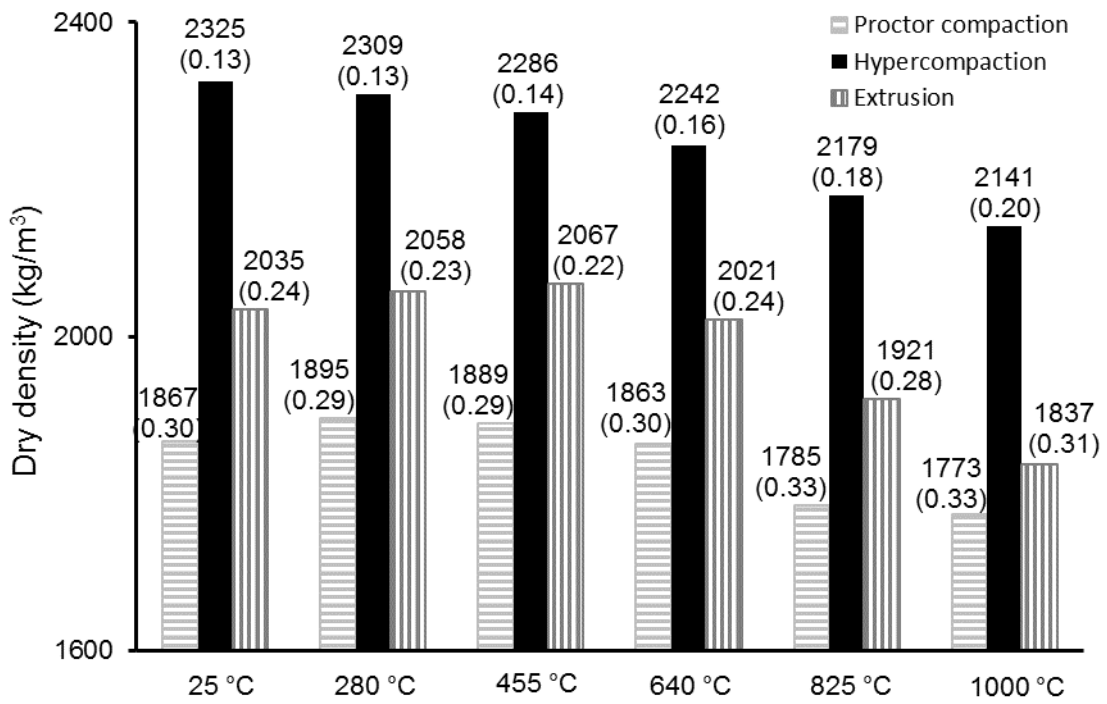


Figure 3. Dry density and porosity (in brackets) of unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.

227 ***TESTING PROCEDURES AND TECHNIQUES***

228 This section presents the laboratory procedures for performing mercury intrusion porosimetry
229 (MIP) tests, compressive strength tests, immersion tests and moisture buffering tests while the
230 corresponding results are discussed in the next section.

231 ***Mercury intrusion porosimetry test***

232 To help interpretation of the macroscopic material properties, MIP tests were carried out on small
233 specimens (about 2 cm³) taken from the brick core. MIP is a laboratory technique that allows
234 investigation of the microstructure of porous media by measuring pore size distribution, density and
235 specific surface. These microstructural characteristics strongly affect the macroscopic behaviour
236 and, in particular, the strength, water durability and moisture buffering capacity of the material.

237 Prior to MIP tests, the specimens were equalised for about one week inside a climatic chamber at a
238 temperature of 25 °C and a relative humidity of 62% to avoid any fabric difference caused by
239 potentially different environmental conditions. After equalisation, the specimens were freeze-dried
240 to remove all free water from the porous network. This procedure consisted in instantaneously
241 freezing the specimens by dipping them in liquid nitrogen at a temperature of -196 °C until
242 termination of boiling. Instantaneous freezing produces the transformation of pore water into
243 amorphous ice with a negligible increase in volume, thus avoiding disturbance to the material fabric
244 (Romero et al., 1999; Nowamooz and Masrouri, 2010; Sasanian and Newson, 2013). Frozen
245 specimens were then exposed to vacuum at a temperature of -50 °C for at least two days to
246 sublimate the pore ice.

247 The freeze-dried specimens were introduced into a penetrometer, which was then inserted inside the
248 low pressure (compressed air) chamber of a Micromeritics AutoPore IV mercury porosimeter. A
249 vacuum corresponding to an absolute pressure of 50 µmHg was applied for 5 minutes to evacuate
250 air and residual moisture from the porous network. Afterwards, mercury was intruded inside the

251 pores with diameters from 10^5 nm to 10^4 nm by increasing the mercury pressure from 10 kPa to 200
252 kPa (low-pressure stage). The penetrometer was then transferred to the high pressure (compressed
253 oil) chamber where the mercury pressure was further increased to 200 MPa to detect the smallest
254 pores down to 10 nm.

255 *Compressive strength test*

256 Compressive strength tests were conducted by using a displacement-controlled Zwick/Roell Amsler
257 HB250 press with a capacity of 250 kN. Bricks were loaded along the longest dimension with a
258 constant displacement rate of 0.001 mm/s (Figure 4). This set-up corresponds to a sample
259 slenderness ratio (i.e. the ratio between the side parallel to the loading direction and the smallest
260 side of the perpendicular cross section) of 4.4 for the extruded bricks and 4 for the Proctor
261 compacted and hypercompacteds bricks. In general, a slenderness ratio bigger than 2 is sufficient to
262 eliminate the effect of spurious confinement owed to end-friction between the brick faces and the
263 press plates. The slightly different slenderness ratio of extruded and compacted bricks should
264 therefore have a negligible effect on the measured strength. End-friction confinement was further
265 reduced by applying Teflon spray on the top and bottom press plates before placing them in contact
266 with the brick extremities and starting the test.

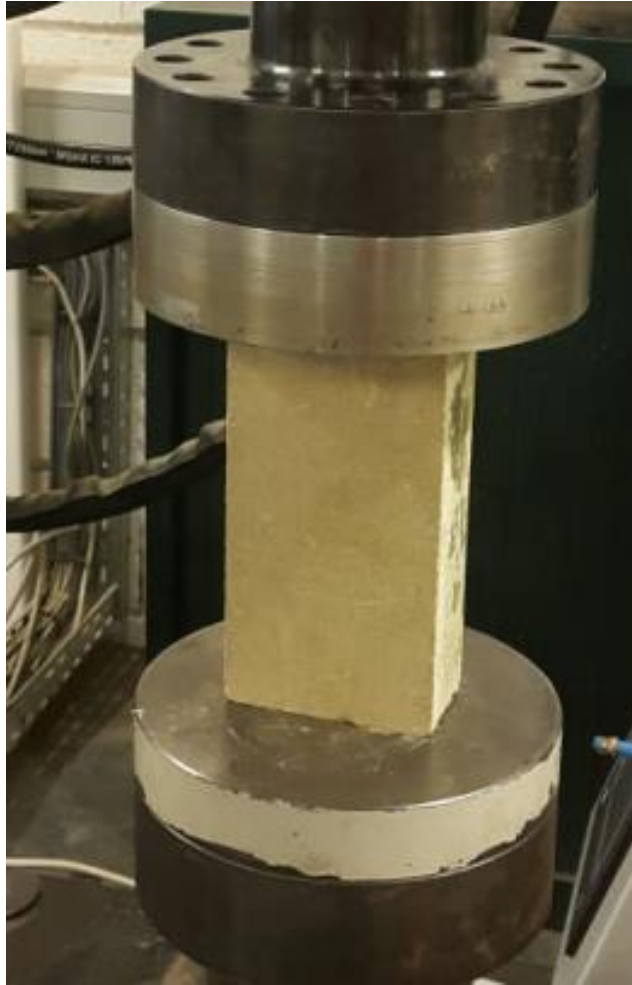


Figure 4. Compressive strength test set-up.

Water immersion test

Water durability was assessed by means of immersion tests in agreement with the norm DIN 18945 (2013). These tests consist in submerging the brick in water for ten minutes and measuring the corresponding mass loss. Prior to immersion, all bricks were equalised to the laboratory atmosphere (temperature of 25 °C and relative humidity of 40%) until a constant mass was achieved and, in any case, for not less than two weeks. After immersion, the bricks were again equalised to the laboratory atmosphere to allow evaporation of adsorbed water and subsequently weighted to determine the mass loss.

278 *Moisture buffering capacity test*

279 A last set of tests was performed to investigate the moisture buffering capacity of the bricks
280 according the norm ISO 24353 (2008). These tests consisted in exposing the bricks to relative
281 humidity cycles inside the climatic chamber CLIMATS (Type EX2221-HA) while simultaneously
282 recording their mass change using a scale with a resolution of 0.01 grams. Prior to the test, the brick
283 surface was sealed with aluminium tape except for one of the two largest faces, which was left
284 exposed to the atmosphere of the climatic chamber. The exposed area was therefore 200 x 100 mm²
285 for Proctor compacted and hypercompact bricks and 220 x 110 mm² for extruded bricks.

286 At the beginning of the test, the bricks were equalised at the lower humidity level of 53% until a
287 constant mass was attained and, in any case, for not less than two weeks. Five relative humidity
288 cycles were then carried out at a constant temperature of 23 °C between the two relative humidity
289 levels of 75% and 53%, with each level maintained for 12 hours. This was sufficient to achieve
290 steady state conditions corresponding to the attainment of a “stable cycle” where moisture uptake at
291 the higher humidity of 75% is identical to moisture release at the lower humidity of 53%. In all tests
292 performed in the present work, the last three cycles were classified as stable cycles.

293 Results from the above test are typically presented in terms of a single parameter, the Moisture
294 Buffering Value (MBV), which is the average mass change Δm (in grams) over the last three stable
295 cycles divided by the exposed sample surface, S (in m²) and the difference between the imposed
296 humidity levels, $\Delta \%RH$ (in %):

$$MBV = \frac{\Delta m}{S \Delta \%RH} \quad (1)$$

297 *RESULTS AND DISCUSSION*

298 This section discusses the results from the above tests comparing microstructure, strength, water
299 durability and moisture buffering characteristics of the different brick types.

300 *Mercury intrusion porosimetry test results*

301 Figure 5 shows the pore size distribution of hypercompacted bricks quickly fired at different
302 temperatures. Note that the unfired material corresponds to the temperature of 25 °C, which is the
303 ambient temperature during equalisation to the laboratory atmosphere. Inspection of Figure 5
304 indicates that the pore size distribution remains virtually unchanged when the firing temperature
305 increases from ambient conditions to 455 °C. However, above 455 °C, the pores larger than 100 nm
306 increase while those below 100 nm tend to progressively disappear. This is reflected by a growth of
307 the characteristic pore size to 250 nm and 1000 nm at the two temperatures of 825 °C and 1000 °C,
308 respectively. This augmentation of the coarsest pore fraction is caused by the burn off of
309 carbonaceous organic matter and the dihydroxylation of structured water above 550 °C, with the
310 consequent formation of sacks of carbon dioxide and water vapour inside the material (Karaman et
311 al., 2006; Baccour et al., 2009; Mahmoudi et al., 2017). This phenomenon is facilitated by the rapid
312 vitrification of the brick surface during quick firing, which creates an impermeable skin impeding
313 evacuation of gases from the brick core.

314 The progressive disappearance of the finest pores at higher firing temperatures has an important
315 impact on the moisture buffering capacity of the material, which is directly related to the amount of
316 pores with sizes of the order of nanometers. This partly explains why firing at higher temperatures
317 entails a progressive loss of the hygro-thermal inertia of the material (McGregor et al., 2016), as
318 shown later in the paper.

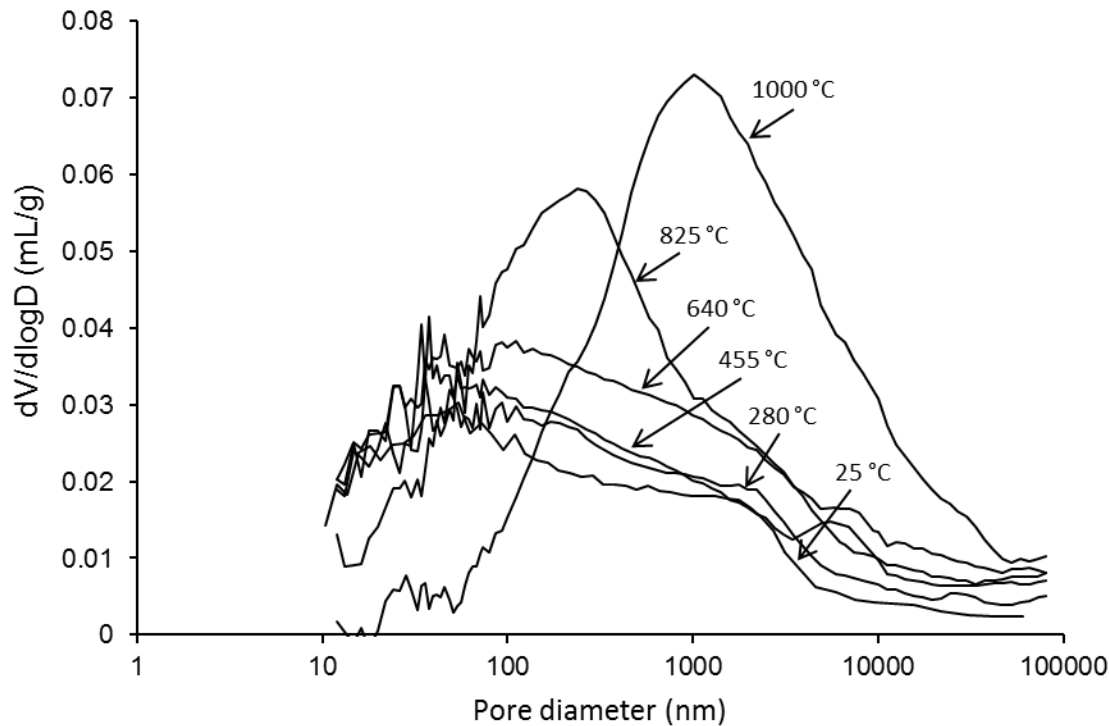
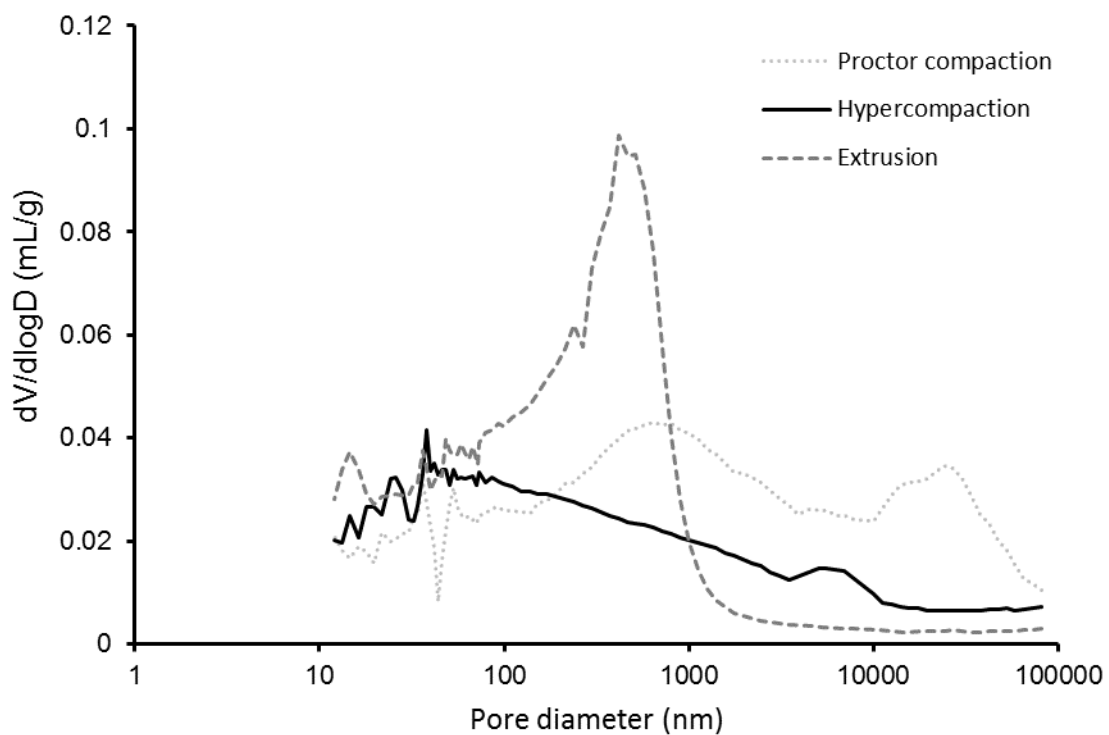


Figure 5. Pore size distributions of hypercompacted unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.

Additional MIP tests were performed on Proctor compacted and extruded bricks quickly fired at 455 °C to investigate the effect of the manufacturing method on the microstructural characteristics. The temperature of 455 °C was selected because, as shown later, this was the lowest temperature at which all bricks, regardless of manufacturing method, exhibit good water durability together with an excellent capacity to buffer moisture. Figure 6 compares the pore size distribution of extruded, Proctor compacted and hypercompacted bricks quickly fired at 455 °C. Differences are evident for the largest pore fraction with diameters bigger than 100 nm while, below 100 nm, the pore size distribution becomes similar for all bricks. The ability of the material to store/release vapour is governed by the finest voids, so the similarity of pore size distributions below 100 nm produces comparable levels of moisture buffering capacity for all bricks, as shown later in the paper.

333 Extruded bricks exhibit a homogenous pore size distribution with a well-defined peak at 500 nm.
 334 On the contrary, Proctor compacted and hypercompact bricks show a heterogeneous porous
 335 network with the consistent presence of different pore diameters. This is partly because, in the case
 336 of extruded bricks, the base earth was ground and passed through a 1 mm sieve, which produces
 337 greater homogeneity of particle sizes compared to Proctor compacted and hypercompact bricks.
 338 This more homogeneous pore size distribution, together with the fact that extrusion at high water
 339 content orients clay platelets along the direction of squeezing, results in better sealing of the outer
 340 surface.



341

342 *Figure 6. Pore size distributions of Proctor compacted, hypercompact*
 343 *and extruded bricks quickly fired at 455 °C.*

344 ***Compressive strength test results***

345 Figure 7 presents the results from compressive strength tests and shows that hypercompact bricks
 346 exhibit significantly higher strength than Proctor compacted and extruded bricks at all firing

temperatures, which is consistent with their greater density (Figure 3). For hypercompacted bricks, quick firing at a relatively low temperature of 455 °C is already enough to attain a very high strength of 29.1 MPa, which is better than current recommendations for masonry buildings exposed to severe weathering (ASTM C62-13a, 2013). The strength of hypercompacted bricks increases even further to 53.1 MPa, a value typical of top performing materials such as high-strength concretes, after quick firing at 825 °C.

Inspection of Figure 7 also indicates that, regardless of the manufacturing method, strength increases as firing temperature rises from 25 °C to 825 °C but then decreases as temperature further grows to 1000 °C. This is in contradiction with previous studies (Karaman et al., 2006; Mbumbia and de Wilmars, 2002) where strength always increased with growing temperature. Comparison of Figures 3 and 7 also indicates that, contrary to unfired earth, strength does not always increase with growing density. These apparently surprising observations are explained by the occurrence of distinct counteracting mechanisms during firing. The first mechanism consists in the almost simultaneous occurrence, at temperatures above 550 °C, of carbonaceous organics burn off and mineral dihydroxylation with the consequent bonding of alumina and silica particles that augments material strength (West and Gray, 1958). This increase of strength is however counteracted by a second mechanism, which is typical of quick firing and consists in the rapid vitrification of the brick surface impeding evacuation of carbon dioxide and water vapour from the inner material. This promotes the formation of large pores with a consequent reduction of density and strength at higher temperatures (Karaman et al., 2006; Baccour et al., 2009). Finally, an increase in temperature above 950 °C induces the transformation of illite (Figure 1) into less stable spinel (MgOAl_2O_3) and hercynite (FeOAl_2O_3) (Jordan et al., 1999 and Aras, 2004), which also contributes to the drop of strength at 1000 °C.

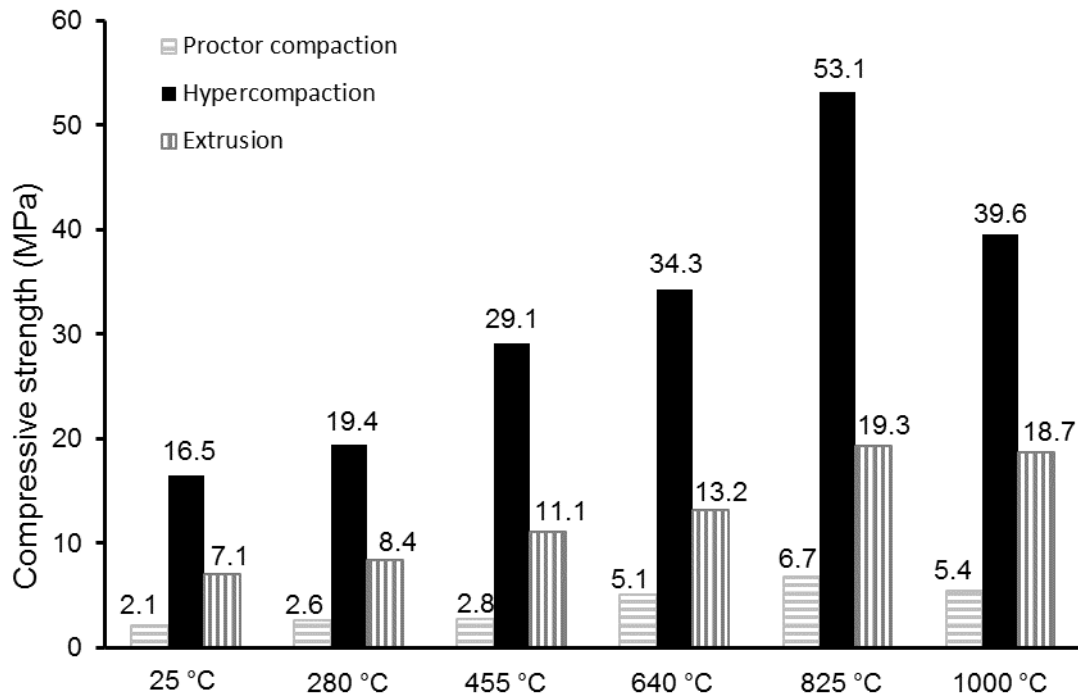


Figure 7. Unconfined compressive strength of unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.

Water immersion test results

A preliminary assessment of water durability was performed by means of immersion tests as prescribed by the norm DIN 18945 (2013). Figure 8 shows the results from these tests in terms of material loss measured after water immersion of Proctor compacted, hypercompacted and extruded bricks quickly fired at different temperatures. Inspection of Figure 8 indicates that, at temperatures smaller or equal to 455 °C, extruded bricks are more durable than Proctor compacted and hypercompacted bricks due to their stronger fabric orientation, which seals the surface and reduces water infiltration. These differences however disappear at temperatures greater than 455 °C, when all bricks exhibit negligible mass loss regardless of the manufacturing method. This indicates that a good water durability might be achieved by firing at significantly lower temperatures and for considerably shorter times compared to current bricks production. Further durability tests, based on complementary experimental protocols, are however necessary to corroborate this conclusion.

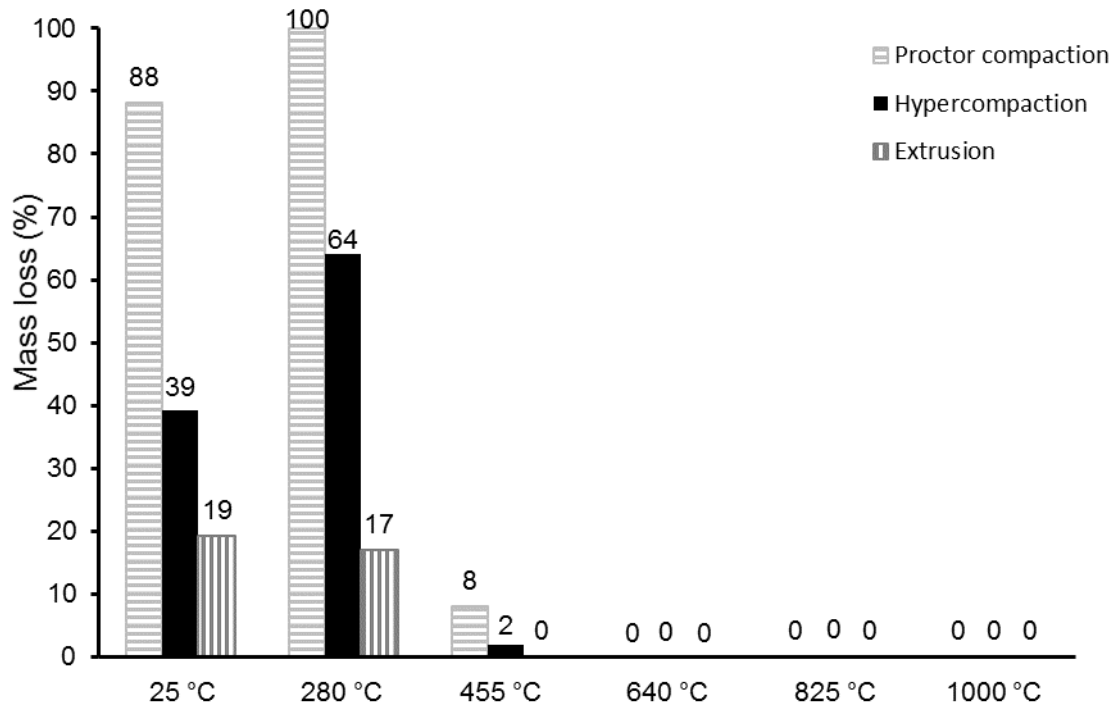


Figure 8. Mass loss after immersion of unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.

Moisture buffering capacity test results

One of the most advantageous properties of raw earth walls is the high hygro-thermal inertia and consequent ability of buffering fluctuations of indoor humidity and temperature. This property originates from the open nanoporous network and high specific surface of the material, which favours the adsorption/release of water vapour together with the simultaneous liberation/storage of latent heat (McGregor et al., 2016). In this respect, the MIP tests presented earlier in this section have shown that the process of quick firing can produce a significant change of pore size distribution, which can in turn influence the moisture buffering capacity of the material.

To further investigate this aspect, moisture buffering tests were performed according to the experimental procedures described in the previous section. The Moisture Buffering Values (MBV) of Proctor compacted, hypercompacted and extruded bricks, quickly fired at different temperatures, are plotted in Figure 9 together with the classification proposed by Rode et al. (2005). Note that this

400 classification is based on an asymmetric humidity cycle of 16h and 8h between 33% and 75%,
401 which is slightly different from the testing procedure adopted in the present work.

402 Inspection of Figure 9 indicates that Proctor compacted bricks exhibit slightly higher moisture
403 buffering capacity compared to hypercompacted and extruded bricks at all firing temperatures. This
404 is justified by the larger porosity of Proctor compacted bricks, which facilitates the exchange of
405 water vapour with the surrounding atmosphere.

406 Inspection of Figure 9 also indicates that the moisture buffering capacity drastically reduces, for all
407 manufacturing methods, as firing temperature increases. This is due to both the progressive
408 vitrification of the brick surface, which reduces the permeability to vapour, and the progressive
409 disappearance of the finest pore fraction, i.e. the fraction smaller than 100 nm, as discussed earlier
410 in the paper (Figure 5). This result is also in agreement with previous works (Mbumbia et al. 2000;
411 Karaman et al., 2006), which observed a progressive reduction of the material capacity to adsorb
412 water vapour with increasing firing temperature. Figure 9 also shows that, at the highest
413 temperature of 1000 °C, the moisture buffering capacity of the material becomes almost negligible.
414 This indicates that the innate ability of raw earth to buffer moisture almost disappears as the firing
415 temperature approaches the levels imposed during the manufacture of commercial bricks.

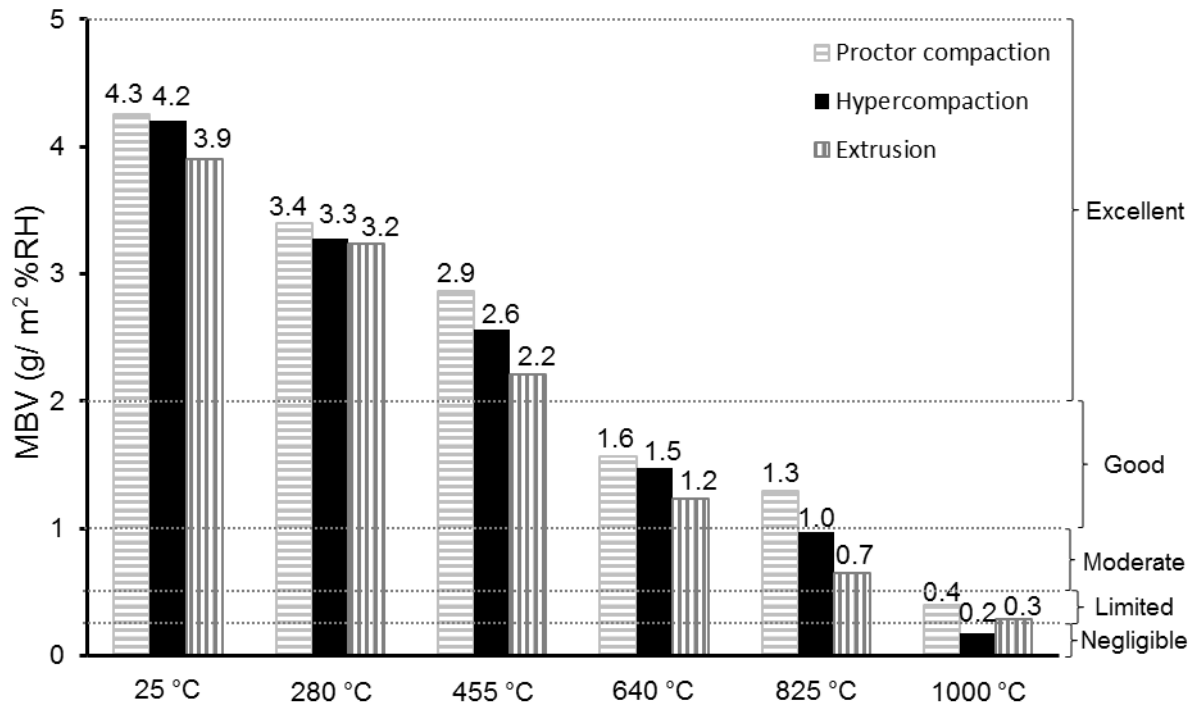


Figure 9. Moisture Buffering Value (MBV) of unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.

Evaluation of proposed manufacturing method

The above results indicate that hypercompacted bricks, quickly fired at a moderate temperature in the range 455 °C - 640 °C, provide the best balance between energy consumption and material properties such as compressive strength (Figure 7), water durability (Figure 8) and moisture buffering capacity (Figure 9).

Table 1 compares the strength, mass loss and moisture buffering value of hypercompacted bricks, quickly fired at 455 °C, with the corresponding values of standard commercial bricks taken from the literature (Brick Industry Association, 2006; Rode et al., 2005). Table 1 also compares the corresponding firing temperatures and times to highlight the advantages of quickly fired hypercompacted bricks in terms of energy costs and production speed. Note that firing time has a different meaning for hypercompacted and standard bricks. In the former case, it indicates the time

430 to attain the desired temperature target while, in the latter case, it indicates the time during which
431 the maximum temperature is maintained.

432 Inspection of Table 1 shows that quickly fired hypercompacted bricks exhibit better compressive
433 strength and moisture buffering capacity than standard bricks. Remarkably, this improvement is
434 attained with lower firing temperatures and times, which also allows a saving of energy, time and
435 carbon emissions. Only water durability is marginally worse for the quickly fired hypercompacted
436 bricks compared to standard ones.

Table 1. Comparison between standard fired bricks and quickly fired hypercompacted bricks

| | Compressive strength (MPa) | Mass loss (%) | MBV (g/m ² %RH) | Firing time (h) | Firing temperature (°C) |
|--------------------------|-------------------------------|------------------|-------------------------------|------------------------|----------------------------|
| Standard fired bricks | 27.0 | 0 | 0.2 | Between 10 and 40 | 1100 |
| Hypercompacted bricks | 29.1 | 2 | 2.6 | 0.67 | 455 |
| Variation (%) | +7.8 | - | +1200 | Between -93 and -98 | -59 |

437

438 **CONCLUSIONS**

439 This paper has presented an innovative and energy-efficient thermo-mechanical process for the
440 manufacture of masonry bricks. The proposed process combines “hypercompaction” of raw earth at
441 a large pressure of 100 MPa with quick firing at low temperatures and times. The process relies on
442 the hypercompaction of raw earth, to generate high levels of material strength, and on subsequent
443 quick firing, to achieve good water durability. A series of laboratory tests was performed to assess
444 the pore fabric, compressive strength, water durability and moisture buffering capacity of
445 hypercompacted bricks quickly fired at five different temperatures of 280, 455, 640, 825 and 1000
446 °C. For comparison, the same properties were also measured on conventional extruded bricks and

447 Proctor compacted bricks subjected to the same thermal treatment. The main outcomes of the
448 research can be summarised as follows:

- 449 • Material strength depends markedly on the manufacturing method with hypercompacted
450 bricks exhibiting the highest strength at all firing temperatures followed by extruded bricks
451 and finally Proctor compacted bricks. This result indicates a direct link between earth
452 densification prior to firing and material strength.
- 453 • The highest strength is always attained at the intermediate firing temperature of 825 °C,
454 rather than at the highest one of 1000 °C. This is a consequence of the fast thermal ramp that
455 is imposed to the earth during quick firing. The highest strength is equal to 6.7 MPa for
456 Proctor compacted bricks, 19.3 MPa for extruded bricks and 53.1 MPa for hypercompacted
457 bricks. This last value is comparable to that of top performing construction materials such as
458 high-strength concretes.
- 459 • Mass loss during water immersion decreases with increasing firing temperatures and
460 becomes negligible above 455 °C for all manufacturing methods. This indicates that
461 adequate water durability can be achieved with significantly lower firing temperatures and
462 times than those adopted during current brick production.
- 463 • Moisture buffering capacity reduces with growing firing temperature in a similar fashion for
464 all manufacturing methods. In particular, bricks fired at a temperature of 1000 °C (i.e. a
465 temperature similar to that imposed during production of commercial bricks) exhibit almost
466 no ability to exchange vapour with the surrounding environment.
- 467 • Based on the above results, quick firing of hypercompacted bricks at relatively low
468 temperatures, between 455 °C and 640 °C, provides the best balance between manufacturing
469 energy and material properties (strength, water durability and moisture buffering capacity).
470 At a temperature of 455 °C, hypercompacted bricks exhibit a strength a 29.1 MPa, a value
471 greater than that recommended by masonry construction guidelines (ASTM C62-13a, 2013).

They also exhibit excellent moisture buffering capacity and almost no mass loss after water immersion.

- Quick firing of hypercompacted bricks at temperatures lower than 455 °C produces negligible changes of pore size distribution with respect to unfired bricks. Above this temperature, however, the material exhibits a progressive augmentation of the coarse pore fraction (i.e. larger than 100 nm) accompanied by a decrease of the fine pore fraction (i.e. smaller than 100 nm). Given that the material ability to store water vapour is directly linked to the extent of the nanoporous network, this observation explains the decrease of moisture buffering capacity with growing firing temperature.
- Extruded bricks present the most uniform porous network with a characteristic size of 500 nm. On the contrary, Proctor compacted and hypercompacted bricks exhibit a relatively heterogeneous porous network with a continuous range of different pore sizes.

The above preliminary results suggest that brickwork factories have the opportunity to improve production quality while significantly reducing manufacturing time, energy consumption and environmental impact. Additional experimental evidence is however necessary to validate the proposed thermo-mechanical brick production process before implementing it at the industrial scale.

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