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	ACCEPTED MANUSCRIPT						
1	OPTIMIZATION OF BRICKS PRODUCTION BY EARTH						
2	HYPERCOMPACTION PRIOR TO FIRING						
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23 HIGHLIGHTS

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

30 ABSTRACT

31 This paper presents an innovative method for the production of masonry bricks, which combines earth compaction and quick firing at low temperatures. Earth bricks were manufactured according 32 to three different methods, i.e. extrusion, standard Proctor compaction and hypercompaction to 100 33 MPa. All bricks were fired inside an electrical furnace by rising the temperature at a quick rate of 34 about 9 °C per minute to 280, 455, 640, 825 and 1000 °C, after which the furnace was turned off 35 and left to cool to the atmosphere with the brick inside it. These firing temperatures and times are 36 significantly lower than those employed for the manufacture of commercial bricks, which are 37 typically exposed to a maximum of 1100 °C for at least 10 hours (Brick Industry Association, 38 2006). A testing campaign was performed to investigate the effect of quick firing on the porosity, 39 40 strength, water durability and moisture buffering capacity of the different bricks. Quick firing of hypercompacted bricks at moderate temperatures, between 455 and 640 °C, is enough to attain very 41 high levels of compressive strength, between 29 and 34 MPa, with a good to excellent moisture 42 buffering capacity. These properties are better than those of commercially available bricks. The 43 strength of hypercompacted bricks further increases to 53 MPa, a value similar to that of high-44 strength concrete, after quick firing at 825 °C. Earth densification prior to thermal treatment 45 therefore improves material performance while enabling a significant reduction of firing 46 temperatures and times compared to current bricks production methods. 47

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49 KEYWORDS

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

52

53 INTRODUCTION

54 Fired earth bricks are commonly employed for the construction of masonry structures despite their relatively large energy and carbon footprints. Bricks exhibit large levels of embodied energy 55 because of their production method which consist in subjecting extruded earth blocks to very high 56 temperatures, up to 1100 °C, for a period between 10 and 40 hours (Brick Industry Association, 57 2006; Zhang, 2013; Murmu and Patel, 2018). This energy-intensive thermal treatment is necessary 58 to achieve adequate mechanical and durability characteristics for construction applications. Besides 59 60 high levels of embodied energy, bricks also exhibit a limited ability to absorb/release vapour from/to the indoor environment, which reduces the hygro-thermal inertia of buildings walls and 61 encourages electrical air conditioning of dwellings (Morton et al., 2005; Rode et al., 2005). Finally, 62 upon demolition, fired bricks generate waste that is often disposed in landfills, thus resulting in 63 environmental pollution and loss of land (Bossink and Brouwers, 1996). 64

Most of the above limitations could be overcome by using raw (i.e. unfired) earth bricks, which are 65 manufactured with relatively little energy as shown by Little and Morton (2001) and Morel et al. 66 (2001). Raw earth also exhibits a strong tendency to adsorb vapour from humid environments and 67 to release it into dry environments while simultaneously liberating and storing latent heat thanks to 68 69 an open network of nanopores and the high specific surface of clay particles. This property increases hygro-thermal inertia and helps smoothing daily fluctuations of humidity and temperature 70 inside buildings with a consequent improvement of occupant comfort and an associated reduction of 71 air conditioning needs (Houben and Guillaud, 1989; Allinson and Hall, 2010; Pacheco-Torgal and 72 73 Jalali, 2012; Soudani et al., 2016; Gallipoli et al., 2017; Soudani et al., 2017). Finally, raw earth is an entirely natural material which can be easily recycled or safely disposed into the environment. 74

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

however shown that "hypercompaction" of earth to very high pressures (of the order of hundreds of 77 megapascals) can produce raw bricks with levels of strength and stiffness that are higher than those 78 of standard fired bricks (Bruno et al., 2017; Bruno et al., 2018). This is possible thanks to a 79 densification of the material down to a porosity of about 0.13, a value similar to that of shale rocks 80 (porosity is the ratio between pore volume and total volume). Unfortunately, this large increase in 81 strength and stiffness does not correspond to a similar gain of durability, especially when raw earth 82 comes into contact with liquid water. For this reason, chemical stabilizers such as cement or lime 83 are often added to the earth to improve mechanical characteristics (Walker and Stace, 1997; Bahar 84 et al., 2004; Guettala et al., 2006; Jayasinghe and Kamaladasa, 2007; Kariyawasam and Jayasinghe, 85 2016; Khadka and Shakya, 2016; Venkatarama Reddy et al., 2016; Dao et al., 2018). Unfortunately, 86 the addition of chemical stabilisers reduces the moisture buffering capacity and hygro-thermal 87 inertia of the material (Liuzzi et al., 2013; McGregor et al., 2014; Arrigoni et al., 2017) while 88 89 largely increasing the carbon footprint (Worrell et al., 2001). Alternative stabilisation methods are therefore necessary to improve water durability without increasing the environmental impact of raw 90 earth. In this respect, the application of moderate heat has been considered in a small number of 91 92 studies as a possible stabilisation method but never in association with a high compaction effort. Mbumbia et al. (2000) investigated the hydro-mechanical behaviour of extruded lateritic earth 93 bricks fired at 350, 550, 750, 850 and 975 °C for 4 and 8 hours. They observed that both mechanical 94 and durability properties improve as temperature increases while firing time has only a marginal 95 effect. These findings were further confirmed by Karaman et al. (2006), who fired pressed earth 96 bricks at temperatures ranging from 700 °C to 1100 °C for different times from 2 to 8 hours. They 97 concluded that temperature plays a key role in changing the physical and mechanical properties of 98 the bricks while firing time has little effect. 99

100 The present work investigates, for the first time, a brick manufacturing method that relies on earth 101 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.

109 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 110 cool to the atmosphere with the brick inside it. As shown later, a moderate temperature, between 111 455 °C and 640 °C, is already sufficient to ensure good levels of water durability. For 112 hypercompacted bricks, this moderate temperature is also sufficient to generate a compressive 113 strength of about 30 MPa, which is greater than the strength of most commercial bricks. 114 Remarkably, if the hypercompacted bricks are quickly fired at a higher temperature of 825 °C, 115 which is however still lower than the temperature imposed during current brick production, material 116 strength increases to an extremely high value of 53 MPa. 117

The results obtained in the present work therefore indicate that a faster, cleaner and less energyintensive 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.

123 MATERIAL AND METHODS

124 The earth used in the present work has been provided by the brickwork factory NAGEN from the 125 region of Toulouse (South-West of France) and corresponds to a typical soil for the production of

standard fired bricks. The grain size distribution was determined by both wet sieving and 126 sedimentation in compliance with the norms XP P94-041 (AFNOR, 1995) and NF P 94-057 127 (AFNOR, 1992), respectively, which indicate that the material is composed by 40.8% sand, 42.9% 128 silt and 16.3% clay. The Atterberg limits of the fine fraction (i.e. the soil fraction smaller than 400 129 µm) were determined according to the norm NF P94-051 (AFNOR, 1993), which indicates a liquid 130 limit of 33.0% and a plasticity index of 12.9%. These results classify the material as an inorganic 131 clay of medium plasticity according to the Unified Soil Classification System USCS ASTM D2487-132 11 (2011). Both grain size distribution and plasticity properties also satisfy existing 133 recommendations for compressed earth bricks (e.g. MOPT, 1992; Houben and Guillad, 1994; 134 CRATerre-EAG, 1998; AFNOR, 2001) as discussed by Bruno (2016). Material mineralogy was 135 investigated by means of X-ray diffractometry using an AXIS Nova X-Ray photoelectron 136 spectroscopy (Kratos Analytica). Results from this test showed that the earth used in the present 137 work is mainly composed of quartz, illite and calcite (Figure 1). 138



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Raw earth bricks were manufactured according to three different methods, namely extrusion, standard Proctor compaction and hypercompaction. Both Proctor compacted and hypercompacted bricks had dimensions of 200 x 100 x 50 mm³, while extruded bricks had slightly larger dimensions of 220 x 110 x 50 mm³. This small variation was the consequence of the different sizes of the screw press ejector of the extruded bricks and the compaction mould of Proctor and hypercompacted bricks. A brief description of the three manufacturing processes is given below:

Extrusion. Extruded bricks were manufactured by the brickwork factory NAGEN according
 to the same process used for standard bricks. The dry earth was passed through a grinder and
 sieved to remove grains larger than 1 mm. The sieved earth was subsequently mixed with an
 optimum water content of about 18% and conveyed to a screw extruder with a rectangular
 ejector section of 110 x 50 mm². Finally, the extruded strip was cut into individual bricks
 with length of 220 mm.

Standard Proctor compaction. The dry earth was mixed at the optimum water content of 153 13.5%, which had been previously determined by standard Proctor compaction of samples at 154 different water contents (AFNOR, 1999). The moist earth was stored inside two plastic bags 155 for at least 24 hours to ensure the equalisation of pore water pressures. The equalised earth 156 was subsequently placed inside a stiff rectangular mould, with a horizontal cross section of 157 $200 \times 100 \text{ mm}^2$, and statically compacted to a target height of 50 mm by a piston with a 158 displacement rate of 0.1 mm/s. The amount of earth placed inside the mould was calculated 159 to attain a dry density of 1860 kg/m^3 , which corresponds to the Proctor optimum. 160

Hypercompaction. The dry earth was mixed at the optimum water content of 5.2%, which
 had been previously determined by static compaction to 100 MPa of samples at different
 water contents (Bruno, 2016). The moist earth was stored inside two plastic bags for 24
 hours to ensure equalisation before being compacted to 100 MPa with a rate of 0.17 MPa/s,
 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 166 a horizontal cross section of 200 x 100 mm². The floating mould was supported by internal 167 friction with the lateral surface of the brick. Double compaction is preferable to single 168 compaction because it reduces frictional effects on the lateral brick surface and therefore 169 increases the uniformity of stress and porosity inside the material. Double compaction could, 170 however, only be employed for hypercompacted bricks because, for Proctor compacted 171 bricks, the applied pressure was too low to generate enough lateral friction to support the 172 weight of the floating mould. Further details about the hypercompaction procedure can be 173 found in Bruno (2016). 174

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

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

As expected, hypercompacted bricks exhibit a higher dry density than Proctor and extruded bricks 197 due to their large compaction pressure. Inspection of Figure 3 also indicates that, for all brick types, 198 dry density decreases as firing temperature grows, especially beyond 455 °C. This result is in 199 contradiction with previous studies (e.g. Karaman et al., 2006) where dry density increased 200 monotonically with growing firing temperatures, which is explained by the quick temperature ramp 201 imposed to bricks in the present work. Quick firing, combined with the high quartz content of the 202 base earth (Figure 1), promotes a rapid vitrification of the brick surface (Cultrone et al., 2004). This 203 impermeable skin then causes the formation of internal "sacks" of carbon dioxide and water vapour 204 with a consequent increase of porosity. Instead, in earlier studies by Karaman et al. (2006) and 205 Mbumbia et al. (2000), a very slow heating rate of only 1°C per minute was applied, which 206 prevented the rapid formation of a vitrified skin and therefore facilitated the evacuation of carbon 207 dioxide and water vapour from the brick core during firing. Note that carbon dioxide and water 208 vapour are typically generated by the burn off of carbonaceous organic matter and the 209 dihydroxylation of structured water at temperatures higher than 550 °C (Karaman, 2006; Baccour et 210 al., 2009). 211

212 Quickly fired bricks were then tested to measure compressive strength, water durability and 213 moisture buffering capacity. Mercury intrusion porosimetry tests were also undertaken to analyse 214 the influence of quick firing on material fabric.

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

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222 TESTING PROCEDURES AND TECHNIQUES

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

226 Mercury intrusion porosimetry test

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

Prior to MIP tests, the specimens were equalised for about one week inside a climatic chamber at a 232 temperature of 25 °C and a relative humidity of 62% to avoid any fabric difference caused by 233 potentially different environmental conditions. After equalisation, the specimens were freeze-dried 234 to remove all free water from the porous network. This procedure consisted in instantaneously 235 freezing the specimens by dipping them in liquid nitrogen at a temperature of -196 °C until 236 termination of boiling. Instantaneous freezing produces the transformation of pore water into 237 amorphous ice with a negligible increase in volume, thus avoiding disturbance to the material fabric 238 (Romero et al., 1999; Nowamooz and Masrouri, 2010; Sasanian and Newson, 2013). Frozen 239 specimens were then exposed to vacuum at a temperature of -50 °C for at least two days to 240 sublimate the pore ice. 241

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

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

250 Compressive strength test

Compressive strength tests were conducted by using a displacement-controlled Zwick/Roell Amsler 251 HB250 press with a capacity of 250 kN. Bricks were loaded along the longest dimension with a 252 constant displacement rate of 0.001 mm/s (Figure 4). This set-up corresponds to a sample 253 slenderness ratio (i.e. the ratio between the side parallel to the loading direction and the smallest 254 side of the perpendicular cross section) of 4.4 for the extruded bricks and 4 for the Proctor 255 compacted and hypercompacted bricks. In general, a slenderness ratio bigger than 2 is sufficient to 256 eliminate the effect of spurious confinement owed to end-friction between the brick faces and the 257 press plates. The slightly different slenderness ratio of extruded and compacted bricks should 258 259 therefore have a negligible effect on the measured strength. End-friction confinement was further reduced by applying Teflon spray on the top and bottom press plates before placing them in contact 260 with the brick extremities and starting the test. 261



Figure 4. Compressive strength test set-up.

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265 *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.

273 Moisture buffering capacity test

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

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

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

$$MBV = \frac{\Delta m}{S \ \Delta \% RH} \tag{1}$$

292 **RESULTS AND DISCUSSION**

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

295 Mercury intrusion porosimetry test results

Figure 5 shows the pore size distribution of hypercompacted bricks quickly fired at different 296 temperatures. Note that the unfired material corresponds to the temperature of 25 °C, which is the 297 ambient temperature during equalisation to the laboratory atmosphere. Inspection of Figure 5 298 indicates that the pore size distribution remains virtually unchanged when the firing temperature 299 increases from ambient conditions to 455 °C. However, above 455 °C, the pores larger than 100 nm 300 increase while those below 100 nm tend to progressively disappear. This is reflected by a growth of 301 the characteristic pore size to 250 nm and 1000 nm at the two temperatures of 825 °C and 1000 °C, 302 respectively. This augmentation of the coarsest pore fraction is caused by the burn off of 303 carbonaceous organic matter and the dihydroxylation of structured water above 550 °C, with the 304 consequent formation of sacks of carbon dioxide and water vapour inside the material (Karaman et 305 al., 2006; Baccour et al., 2009; Mahmoudi et al., 2017). This phenomenon is facilitated by the rapid 306 vitrification of the brick surface during quick firing, which creates an impermeable skin impeding 307 308 evacuation of gases from the brick core.

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

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Additional MIP tests were performed on Proctor compacted and extruded bricks quickly fired at 318 455 °C to investigate the effect of the manufacturing method on the microstructural characteristics. 319 The temperature of 455 °C was selected because, as shown later, this was the lowest temperature at 320 which all bricks, regardless of manufacturing method, exhibit good water durability together with 321 an excellent capacity to buffer moisture. Figure 6 compares the pore size distribution of extruded, 322 323 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 324 distribution becomes similar for all bricks. The ability of the material to store/release vapour is 325 governed by the finest voids, so the similarity of pore size distributions below 100 nm produces 326 comparable levels of moisture buffering capacity for all bricks, as shown later in the paper. 327

328 Extruded bricks exhibit a homogenous pore size distribution with a well-defined peak at 500 nm. 329 On the contrary, Proctor compacted and hypercompacted bricks show a heterogeneous porous network with the consistent presence of different pore diameters. This is partly because, in the case 330 of extruded bricks, the base earth was ground and passed through a 1 mm sieve, which produces 331 greater homogeneity of particle sizes compared to Proctor compacted and hypercompacted bricks. 332 This more homogeneous pore size distribution, together with the fact that extrusion at high water 333 content orients clay platelets along the direction of squeezing, results in better sealing of the outer 334 surface. 335





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Figure 7 presents the results from compressive strength tests and shows that hypercompacted bricksexhibit 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 348 349 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 350 and de Wilmars, 2002) where strength always increased with growing temperature. Comparison of 351 Figures 3 and 7 also indicates that, contrary to unfired earth, strength does not always increase with 352 growing density. These apparently surprising observations are explained by the occurrence of 353 distinct counteracting mechanisms during firing. The first mechanism consists in the almost 354 simultaneous occurrence, at temperatures above 550 °C, of carbonaceous organics burn off and 355 mineral dihydroxylation with the consequent bonding of alumina and silica particles that augments 356 material strength (West and Gray, 1958). This increase of strength is however counteracted by a 357 second mechanism, which is typical of quick firing and consists in the rapid vitrification of the 358 brick surface impeding evacuation of carbon dioxide and water vapour from the inner material. This 359 360 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 361 950 °C induces the transformation of illite (Figure 1) into less stable spinel (MgOAl₂O₃) and 362 hercynite (FeOAl₂O₃) (Jordan et al., 1999 and Aras, 2004), which also contributes to the drop of 363 strength at 1000 °C. 364





368 Water immersion test results

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A preliminary assessment of water durability was performed by means of immersion tests as 369 prescribed by the norm DIN 18945 (2013). Figure 8 shows the results from these tests in terms of 370 material loss measured after water immersion of Proctor compacted, hypercompacted and extruded 371 bricks quickly fired at different temperatures. Inspection of Figure 8 indicates that, at temperatures 372 smaller or equal to 455 °C, extruded bricks are more durable than Proctor compacted and 373 hypercompacted bricks due to their stronger fabric orientation, which seals the surface and reduces 374 375 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 376 good water durability might be achieved by firing at significantly lower temperatures and for 377 considerably shorter times compared to current bricks production. Further durability tests, based on 378 complementary experimental protocols, are however necessary to corroborate this conclusion. 379



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383 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

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

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

Inspection of Figure 9 also indicates that the moisture buffering capacity drastically reduces, for all 401 manufacturing methods, as firing temperature increases. This is due to both the progressive 402 vitrification of the brick surface, which reduces the permeability to vapour, and the progressive 403 disappearance of the finest pore fraction, i.e. the fraction smaller than 100 nm, as discussed earlier 404 in the paper (Figure 5). This result is also in agreement with previous works (Mbumbia et al. 2000; 405 406 Karaman et al., 2006), which observed a progressive reduction of the material capacity to adsorb water vapour with increasing firing temperature. Figure 9 also shows that, at the highest 407 temperature of 1000 °C, the moisture buffering capacity of the material becomes almost negligible. 408 This indicates that the innate ability of raw earth to buffer moisture almost disappears as the firing 409 temperature approaches the levels imposed during the manufacture of commercial bricks. 410



412 Figure 9. Moisture Buffering Value (MBV) of unfired (25 °C) and quickly fired (280, 455, 640, 825, 1000 °C) bricks.
 413

414 Evaluation of proposed manufacturing method

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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

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

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

	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			

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

432

433 CONCLUSIONS

This paper has presented an innovative and energy-efficient thermo-mechanical process for the 434 manufacture of masonry bricks. The proposed process combines "hypercompaction" of raw earth at 435 a large pressure of 100 MPa with quick firing at low temperatures and times. The process relies on 436 the hypercompaction of raw earth, to generate high levels of material strength, and on subsequent 437 quick firing, to achieve good water durability. A series of laboratory tests was performed to assess 438 the pore fabric, compressive strength, water durability and moisture buffering capacity of 439 440 hypercompacted bricks quickly fired at five different temperatures of 280, 455, 640, 825 and 1000 °C. For comparison, the same properties were also measured on conventional extruded bricks and 441

442 Proctor compacted bricks subjected to the same thermal treatment. The main outcomes of the443 research can be summarised as follows:

Material strength depends markedly on the manufacturing method with hypercompacted
 bricks exhibiting the highest strength at all firing temperatures followed by extruded bricks
 and finally Proctor compacted bricks. This result indicates a direct link between earth
 densification prior to firing and material strength.

The highest strength is always attained at the intermediate firing temperature of 825 °C,
rather than at the highest one of 1000 °C. This is a consequence of the fast thermal ramp that
is imposed to the earth during quick firing. The highest strength is equal to 6.7 MPa for
Proctor compacted bricks, 19.3 MPa for extruded bricks and 53.1 MPa for hypercompacted
bricks. This last value is comparable to that of top performing construction materials such as
high-strength concretes.

Mass loss during water immersion decreases with increasing firing temperatures and
 becomes negligible above 455 °C for all manufacturing methods. This indicates that
 adequate water durability can be achieved with significantly lower firing temperatures and
 times than those adopted during current brick production.

Moisture buffering capacity reduces with growing firing temperature in a similar fashion for
 all manufacturing methods. In particular, bricks fired at a temperature of 1000 °C (i.e. a
 temperature similar to that imposed during production of commercial bricks) exhibit almost
 no ability to exchange vapour with the surrounding environment.

Based on the above results, quick firing of hypercompacted bricks at relatively low temperatures, between 455 °C and 640 °C, provides the best balance between manufacturing energy and material properties (strength, water durability and moisture buffering capacity).
At a temperature of 455 °C, hypercompacted bricks exhibit a strength a 29.1 MPa, a value greater than that recommended by masonry construction guidelines (ASTM C62-13a, 2013).

- 467 They also exhibit excellent moisture buffering capacity and almost no mass loss after water468 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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